Agency for Toxic Substances and Disease Registry
Division of Health Studies

FINAL REPORT
Technical Assistance to the Illinois Department of Public Health

Exposure Study of Volatile Organic Compounds Southeast Rockford, Illinois

October 1991

U.S. DEPARTMENT OF HEALTH & HUMAN SERVICES
Public Health Service
Agency for
Toxic Substances and Disease Registry
Atlanta, Georgia 30333

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ABSTRACT

In September 1989 the Illinois Department of Public Health (IDPH) requested technical assistance from the Agency for Toxic Substances and Disease Registry (ATSDR) to evaluate blood levels of volatile organic compounds (VOCs) in residents of southeast Rockford, Illinois, after elevated levels of these compounds were found in the groundwater. A VOC exposure study was conducted on selected residences in which water, air levels, and blood levels of the occupants were measured. The purpose of the study was to determine if 1) the southeast Rockford residents had mean blood levels of VOCs higher than those reported in a subset of the National Health and Nutritional Examination Survey (NHANES III) and 2) a correlation could be established between blood levels and environmental exposures (water and air) in the home.

Study power was greatly reduced since many residents were ineligible for study participation because they voluntarily used bottled water. Four of the ten individuals tested had blood levels of one VOC greater than two standard deviations above the NHANES III mean. While no statistically significant correlations were found between blood and water levels in the study population, several correlations between blood and air levels were statistically significant. For only one compound was a statistically significant association found between air and water measurements. While this investigation suggests that air may be an important route of exposure to VOCs, the study is limited by both very low statistical power and multiple comparisons.

INTRODUCTION

The Illinois Department of Public Health (IDPH), with technical assistance from the Agency for Toxic Substances and Disease Registry (ATSDR), evaluated blood levels of volatile organic compounds (VOCs) in residents of southeast Rockford, Illinois after elevated levels of these compounds were found in drinking water. During the preliminary health assessment done as part of a cooperative agreement with ATSDR at the National Priorities List (NPL) site in southeast Rockford, IDPH found elevated levels of VOCs in the groundwater. IDPH and ATSDR concluded that the site was of imminent public health concern and the study proposed. On October 1 and October 2, 1989, representatives from IDPH met with the Division of Health Studies (DHS) of ATSDR to design a study to evaluate the VOC exposure of residents of the site.

BACKGROUND

Groundwater contamination in Rockford was first identified in 1984 as a result of an investigation by the Illinois Environmental Protection Agency (IEPA) into the dumping activities of an electroplating company. Several nearby residential wells were sampled by the IEPA for compounds characteristic of electroplating activities (chromium, cadmium, and cyanide). Although none of these types of compounds was detected in the wells, several volatile organic compounds, including 1,1,1-trichloroethane (1,1,1-TCA), trichloroethylene (trichloroethene or TCE), cis-1,2-dichloroethylene (cis-1,2-dichloroethene) and 1,1-dichloroethylene (1,1-dichloroethene or DCE), were detected by the IDPH. Subsequent residential well sampling by the IDPH showed that the contamination was widespread. By October 1989, more than 150 private wells and one municipal well had been found to have varying levels of contamination by multiple VOCs, with several private wells having a total VOC concentration in excess of 500 parts per billion (ppb). Table 1 shows the levels of VOCs measured as of October 1989. As of June 1990, the area of contamination covered approximately 2 square miles (bordered to the north by Harrison Avenue, to the east by 21st Street, to the south by Sawyer Avenue, and to the west by 8th Street) (Figure 1). No source for the contamination was identified for the NPL site.

The VOCs of primary health concern are chlorinated hydrocarbons, commonly used as solvents. These chemicals are readily absorbed when inhaled or ingested. Dermal absorption, however, is thought to be poor. The targets of acute and chronic toxicity are primarily the central nervous system (CNS), liver, and kidneys in both humans and animals.[1-3]

Acute CNS effects are proportional to the concentration of chemicals in the blood. All other toxic effects of these chemicals appear to require metabolic alteration. The toxicity of each chemical, therefore, varies with its chemical-specific rate of biotransformation. It also varies with species, strain, and sex.[1-3]

The relationship between length of exposure and acute or chronic symptoms is unclear. Repeated exposure may cause acute episodes of toxicity, and has been implicated in chronic health effects[1-3]

Purpose

The purpose of this study was twofold: (1) to determine if the levels of the predominant VOCs in the blood of study area residents were above mean concentrations reported by the National Health and Nutritional Examination Survey (NHANES III), a national study designed to collect baseline data on a number of health parameters; 2) to determine if a correlation between blood level and environmental level could be detected.

METHODS

Overview

Selection of households from the study area was based on water sampling results, which are characterized in Table 1. To be included in the study, a household's well water had to contain 1) at least 5 ppb trichloroethylene and 100 ppb total VOCs or 2) 500 ppb total VOCs; in addition, the household had to be using the well for drinking water at the time of the study. To minimize potential confounding from occupational exposures, the study targeted persons likely to spend time in their homes: nonpregnant women aged 20 through 65 who worked outside the home no more than 25 hours per week. Only one female per household was included in the study. Once consent was given, the subject was interviewed and a phlebotomist collected a blood sample. If the subject also consented to air monitoring, samples of the ambient household air were taken and an air-sampling questionnaire was administered; this was done either the same day as the interview and blood sampling or the following day.

Rationale for study design

This investigation was designed as a descriptive study to evaluate the presence of VOCs in the blood of subjects whose primary exposure was through the use of contaminated groundwater. Subject selection was structured to optimize the potential exposure of persons selected, while minimizing potential confounding exposures such as occupational exposures or hobbies. No comparison group was selected specifically for this study because background levels of blood VOCs could be obtained from NHANES III.

Area selection

The exposed area was defined as the area in southeast Rockford, Illinois known to have contaminated groundwater as of October 1, 1989.

Sample selection

Based on the results of the water sampling, households were assigned priorities from highest exposure potential to lowest exposure potential and were contacted in that order.

Selection of households eligible for inclusion in the study was based on the analytical results of the most recent well water samples taken by the U.S. Environmental Protection Agency (USEPA) or the IDPH. The criteria were selected so that households sampled would have the greatest potential exposure and so that a sufficient number of households could be contacted to produce a sample of from 30 to 50 eligible subjects.

Once contact was made with a household, information was obtained to determine eligibility. Wells were required to be in current use as drinking water sources by the household, and the household also had to contain a nonpregnant female aged 20 to 65 years. There were no restrictions on the type of employment in which a participant might be engaged. Only women employed fewer than 25 hours per week outside the home were included in the study because many area residents were employed in industries using volatile organic solvents. By excluding persons employed outside the home more than 25 hours a week, potential problems with occupational confounding could be minimized. Additionally, women tend to use household water more often (for washing clothes and dishes and housecleaning) and so would be expected to have a higher exposure potential. Furthermore, women over 65 years of age were not selected in order to minimize possible age-related changes in metabolism of VOCs. Only one female from a given household was included in the study. If any of the criteria were not met, the household was eliminated from further consideration and the next household on the list was contacted.

If all of the criteria were met and the individual agreed to participate in at least the questionnaire and blood-drawing phases of the study, the household was included in the study. If more than one eligible subject was available, the interviewer arranged them in order by age (oldest to youngest) and contacted the study leader for random selection of the participant.

Tracing

Interviewers completed contact forms detailing the numbers and times of attempted contacts with selected subjects, as well as information necessary to establish the eligibility of households for inclusion in the study. In each case, if no contact was made on the first try, two additional visits were made to a house at different times on two different days. Notification letters requesting a resident of the household contact IDPH were left after each unsuccessful visit. Two additional attempts to contact the household by telephone were made at different times on two different days before it was eliminated from consideration.

Interviewing and sampling

Once it was determined that an individual in a household was eligible for inclusion in the study, the interviewer presented an introductory letter from Bernard Turnock, M.D., Director of IDPH at the time and the appropriate consent form (Appendix 1) to the subject. If the individual agreed to participate, she was asked to sign the consent form and given a copy. After the subject completed the consent form, she was interviewed. Following the interview, the interviewer contacted the phlebotomist to arrange blood sampling the same day. A form was left with the participant to be completed by the phlebotomist after the blood sample was taken. At the same time the phlebotomist was contacted, the air sampling team was contacted and arrangements was made to monitor the indoor air. Again, forms were left with the participant for the air sampling team to complete following the air sampling.

Data sources

Questionnaires: Two questionnaires were developed for and used in this study: 1) an individual questionnaire designed to characterize subject exposure and

identify possible confounding exposures and 2) an air sampling questionnaire designed to provide information about the air sampling procedures performed and to verify and supplement the exposure information collected by the individual questionnaire.

The individual questionnaire included questions on general household information: the source of the household water supply, the presence of sump pumps and untrapped drains, the use of humidifiers and all household appliances, the frequency of household duties performed that required water use, water consumption, showering and bathing practices, and exposure to potential confounding factors. Specific confounders studied were: visiting a drycleaners, pumping gasoline, house painting, remodeling, participating in hobbies with potential exposure to VOCs, bringing home work clothes from jobs with potential exposure to VOCs, home laundering of work clothes from jobs with potential exposure to VOCs, and using tobacco products and alcoholic beverages.

The air sampling questionnaire contained questions about the length of time that had elapsed since water had been used for various noncooking purposes, as well as questions about the use of clothes dryers and humidifiers. This questionnaire was designed to characterize the use of water within a household during the time air sampling was done. Copies of the household questionnaire and the air sampling questionnaire are included in Appendix 1.

The questionnaires were adapted from ones used previously by ATSDR. Additional questions were added about potential confounders and the use of household appliances and water within the household.

Pretesting and pilot testing of the questionnaires were not done because the residents of the study area needed to be given alternative sources of water as soon as possible. Since VOCs are rapidly metabolized and eliminated, it was necessary to take blood samples while the subjects were still ingesting contaminated water. The study was planned on October 2 and October 3, 1989, and interviewing was conducted from October 18 through October 21, 1989. By the time the study was designed, households in the study area were scheduled to receive bottled water. Delaying the study further was not feasible because the subjects would no longer be drinking contaminated water and any VOCs that may have been present in the blood would have been eliminated.

Interviewers were trained during a 4-hour orientation session. This session emphasized the need for objective data. Interviewers were told not to deviate from the questionnaire and to avoid words or behavior that would encourage any specific response. Simulated interviews also were conducted during this session. The trainers interviewed each other, and the interviewers recorded the responses they thought were appropriate. These responses were compared with those recorded by the trainers and discrepancies were resolved.

The USEPA air sampling team was trained to administer the air sampling questionnaire. This training involved a separate 2-hour session also emphasizing the need for objective questionnaire data. They, too, were told not to deviate from the questionnaire and to avoid words or behavior that

would encourage any specific responses. Simulated interviews were included in this training session.

The study coordinator checked each completed questionnaire item-by-item upon its return to determine whether it had been completely filled out. Additionally, spot checks on interviewers were conducted to ensure proper procedures were being followed. A sample of persons interviewed were called by the study coordinator and asked to verify the responses recorded by the interviewers.

Blood sampling: Blood samples were drawn from participants by a qualified phlebotomist. Two 10-milliliter (ml) vials of blood were drawn from each participant and stood upright on wet ice (approximately 4 degrees C). The vacutainer tubes were supplied by the Centers for Disease Control, National Center for Environmental Health and Injury Control (CDC, NCEHIC)*, Division of Environmental Health Laboratory Sciences, and were prescreened for VOC contamination. Filled tubes were encoded by a CDC, NCEHIC representative and shipped to the CDC, NCEHIC laboratory in Atlanta, Georgia.

VOCs in whole human blood were analyzed by the Division of Environmental Health Laboratory Sciences of CDC, NCEHIC using a heated purge and trap gas chromatograph/mass spectrophotometer (GC/MS). The protocol used for this analysis is included in Appendix 2. It should be noted that the sensitivity and thus the detection limits for these analyses varied during different sample runs. Therefore, the detection limits in Appendix 2 should be considered average detection limits. Individual runs may have had lower limits.

Air sampling: Indoor air monitoring was conducted in eligible households to determine the relative contribution of volatilized VOCs to overall exposure. Air was sampled to measure both peak and chronic exposures.

1. Utilizing evacuated air bags, samples at 0, 10, and 20 minutes were collected in one bathroom while the shower was running. These samples were analyzed for specific VOCs on a Photovac gas chromatograph. These samples measure peak exposure.

2. Using Tenax tubes and personal sampling pumps, samples were collected during the same sampling periods mentioned previously (#1). These samples were analyzed by GC/MS and also indicate maximum exposure.

3. Using Tenax tubes and personal sampling pumps placed in the breathing zone, samples were collected for 12-hour periods inside and outside each home. These also were analyzed by GC/MS and are a measure of cumulative exposure.

4. Using Tenax tubes and a personal sampling pump, a sample was taken in the yard of one of the study households.

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* National Center for Environmental Health and Injury Control was formerly known as the Center for Environmental Health and Injury Control

The protocol for these analyses is included in Appendix 2.

Study power

If an elevated level is defined as greater than two standard deviations (SD) from the mean (approximately 5% of samples) and 30 to 50 samples are collected, then it would be necessary for 25% of the study samples to have elevated levels to have a statistically significant finding.[4] Consequently, this study has limited power to detect a difference between the study group and the comparison NHANES group.

Data entry and quality control

Results of the blood, air, and water analyses for each study participant were transferred to a coding sheet. The data were transferred exactly as reported by the laboratories and doublechecked for accuracy by a single individual. Data from the air sampling questionnaire and the household/individual questionnaire were entered directly from the questionnaires, using the Epi Info software package developed by CDC, which permits data entry onto screens similar to the data collection forms. All data were entered twice into two separate data sets. The data sets were compared variable by variable and any discrepancies were resolved. This was done by printing each variable for each household and verifying the variable was identical in both data sets. After an edited data set had been created, it was converted to a Statistical Analysis System (SAS) data set, and loaded onto a mainframe system. Subsequent transformations and analyses were performed using SAS.

Data transformations

Blood: Blood measurements were reported as either numerical values or a notation that no chemical had been detected. When a compound was not detected in half or more of the blood samples, the samples for which no chemical had been detected were eliminated from further statistical analyses. When fewer than half of the measurements in a series were below the detection limit, the samples in which no chemical had been detected were assigned a value equal to half the detection limit of the compound and included in subsequent analyses.

Water: For each VOC, results were reported as either measurable (using a numeric value), as not measurable (no chemical detected), or as not quantifiable (chemical present but the level cannot be accurately determined). When half or more of the measurements of a given variable were either not measurable or not quantifiable, only samples with numerical values were included in subsequent analyses. When fewer than half of the measurements in a series were below detection level, the measurements reported as not measurable were assigned a zero value, and the measurements reported as not quantifiable, were assigned a value equal to half the limit of detection.

Air: Results were reported as either as measurable (numerical values), as not measurable (no chemical detected), or as not quantifiable (chemical present but at a level too low to be measured accurately). When half or fewer of the measurements of a given variable were measurable, only those samples with numerical values were included in subsequent analyses. When more than half of the measurements in a series were numerical values, the not measurable samples

were assigned a zero value and the not quantifiable samples were assigned a value equal to half the limit of detection.

When duplicate air measurements were made, a mean value was assigned to the household, with the exception of the measurement of 1,2-dichloroethane, which was taken by Tenax tube when the household shower was running. Only one household had a measurement of 1,2-dichloroethane at a quantifiable level; that measurement was reported separately.

None of the two 12-hour air samples taken from each household were started at the same time of day and, therefore, no single measurement was comparable for all of the households. To obtain a more representative comparison of these samples, the mean of the two samples for each household was used. One of the households was sampled during only one 12-hour period. Adjustments for this household were made in three ways: 1) by eliminating the household from subsequent analyses, 2) by dividing the measurements obtained from this household by two, which minimized the exposure estimates but gave the least possible exposure levels known to have occurred, and 3) by assuming that the 12-hour sample was representative of the 24-hour exposure situation and using it as an approximation of the mean 24-hour level. Correlations with the 24-hour air measurements are presented for all of the ways the adjustment was made for the household with only one 12-hour sample.

Data analyses

Descriptive: Each variable was characterized by the total number of measurements, the number of measurements that were non-detectable, the number of measurements below the detection limits or duplicate measures, the number of measurements included in the statistical analyses, the mean, the median, and the range. For each variable included in the statistical analyses, the number of observations, and the mean, median, and range of values were calculated.

Analytical: Spearman rank correlations for blood levels, water levels, and air measurements were calculated and tested for statistical significance using the 2-sided 0.05 level. Mean blood levels measured in the study population were calculated. The frequency of measurements greater than two standard deviations over the mean reported by the NHANES III survey also was noted.

RESULTS

Comparison of participants and nonparticipants

Sixty-three households were contacted, and 10 eligible individuals agreed to participate in the household interview and blood-drawing phase. Air sampling was done in seven households. Table 2 lists the reasons the 53 households did not participate. Eighteen households were excluded because they were not using their well water for drinking and cooking at the time of the survey. Five households were excluded because they used water filters; 10 households did not have a nonpregnant female aged 20 to 65 years living in residence; and 14 households were excluded because all of the women aged 20 to 65 years worked outside the home more than 25 hours per week. Three households refused to participate in the study and three households could not be contacted.

Table 3 shows the mean levels of TCE and total VOCs measured in the well water of both the 10 participants and the 53 nonparticipants. A two-sided Student's t-test showed that the well water of study participants did not differ significantly from that of nonparticipants with respect to TCE ($p = .74$) or total VOCs ($p = .56$).

Descriptive analyses

The total number of measurements taken for each variable, the number of measurements indicating no chemical had been detected, and the number of indications the chemical was present but at a level that could not be quantified are shown in Table 4. Trans-1,2-dichloroethylene was not detected in measurable levels either in air or in blood samples. Only one of the air measurements of 1,2-dichloroethane was quantifiable; blood levels of this compound were not analyzed. Therefore, further statistical analyses were not performed on trans-1,2-dichloroethylene or 1,2-dichloroethane.

For each variable, the number of observations used, the median, the mean, and the range are shown in Tables 5,6, and 7. The compounds appearing at the highest levels in the blood were 1,1,1-trichloroethane (mean = 1.40 ppb) and tetrachloroethylene (mean = 0.21 ppb). Mean water levels were highest for 1,1,1-trichloroethane (mean = 211.93 ppb) and cis-1,2-dichloroethylene (mean = 76.73 ppb); however, only three measurements were made of cis-1,2-dichloroethylene. Airbag sampling in the shower area yielded quantifiable levels only of trichloroethylene and 1,1-dichloroethylene. The highest mean levels obtained by Tenax tube sampling of shower areas were of 1,1,1-trichloroethane (24.65 ppb) and 1,1-dichloroethane (13.70 ppb). Household air measurements were highest for 1,1,1-trichloroethane and trichloroethylene. Measurements obtained in the backyard and the 12-hour ambient air household measurements for the household with backyard sampling are listed in Table 8. Measurements taken in the backyard were lower than those taken in the household, except for one household measurement of tetrachloroethylene.

Inferential analyses

Correlations between blood, water, and air levels for the compounds studied are shown in Table 9. No statistically significant ($p<0.05$) correlations were found between blood measurements and water measurements. Several statistically significant correlations were found between blood and air measurements. When mean household levels of air concentrations were calculated using the measurements made for only 12 hours with no other adjustment, the Spearman rank correlation of blood and household air levels of tetrachloroethylene was statistically significant ($r = 0.79$, $p = 0.03$). Significant correlations were also found for blood and household air measurements of 1,1-dichloroethane.

For one compound, the association between water and air measurements was statistically significant. The Spearman rank correlation of water levels of 1,1-dichloroethylene was significantly correlated with the Tenax shower measurements ($r = .90$, $p = 0.04$).

Comparisons of the mean blood levels of compounds measured in the study population with the means obtained in the NHANES III survey are shown in

Table 10. Three measurements of cis-1,2-dichloroethylene were greater than two standard deviations above the NHANES III mean used for comparison. One measurement of trichloroethylene was greater than two standard deviations above the NHANES III mean.

Potential Confounders

Tabulations of the confounding variables are shown in Table 11. In general, the 10 subjects tended to be similar with respect to confounders. The most common confounding exposures to volatile organic compounds were due to the occupations of other household members. Seven of the ten subjects had a household member with an occupation involving solvent exposure and each of these subjects reported that workclothes were worn home from these jobs. Five subjects also laundered these workclothes at home. The following confounding factors were reported by three subjects: present consumption of alcohol, current smoking, and household remodeling within one month prior to the study. The subject with the highly elevated trichloroethylene level reported no exposure to the confounders studied.

DISCUSSION

The results of this study provide some limited evidence about the association between exposure to VOCs and elevated levels of such compounds in the blood. Four of the ten participants had a VOC measurement greater than two standard deviations above the mean values reported by the NHANES III study. The correlation found between shower samples and water levels of 1,1-dichloroethylene suggests water is an important source of air exposure to this compound. Blood and air measurements of tetrachloroethylene were correlated when all observations were used and no adjustments were made for sampling time. Blood levels of 1,1-dichloroethane were correlated with household air measurements.

Although some of the results suggest that exposures to VOCs may result in elevated blood levels, other results do not support this association. No direct correlation was found between water levels and blood levels of VOCs and no correlation was found between long-term household air levels of VOCs and blood levels of VOCs. The increases in air levels of VOCs with increasing shower run-time and the correlation between 1,1-dichloroethylene in the shower air and water suggest that water contamination may contribute to elevated air levels. However, since this correlation was statistically significant for only one compound, the link between water contamination and air exposure is only suggested. The mean blood levels of the study population were not consistently greater than those of the NHANES III population. For two of the compounds which occurred at relatively high levels in the water, mean blood levels were lower in the study population than in the NHANES III population. It is noteworthy that most of the elevated blood levels were found for the same compound, cis-1,2-dichloroethylene, and the elevated level of trichloroethylene was one of the highest levels observed to date by the NCEHIC Division of Environmental Health Sciences Laboratory.

This study is the first ATSDR study of water contamination by VOCs that has shown blood levels of cis-1,2-dichloroethylene and of trichloroethylene greater than the reference levels currently used for VOCs. This study yielded more elevated measurements of blood VOCs than did previous studies of blood

VOCs in persons residing in contaminated areas. A study of VOC blood levels of persons living near the Industrial Excess Landfill NPL site in Uniontown, Ohio, found that 2 subjects out of the 13 tested had elevated levels of tetrachloroethylene. However, one of these participants worked in a drycleaning establishment.[5] Another study conducted in Doylestown, Pennsylvania, found no blood levels greater than the reference levels in use at that time.[6]

Several factors limit the inferences that can be drawn from this study. The study sample was not a representative sample from the study area because only households with relatively high VOC levels were included. The control group was not from the Rockford area and data on potential confounding factors were not available for the control group (the NHANES III sample). While the data on potential air exposures were extensive, even more detail would have made it possible to predict more precisely the air levels of VOCs from water levels. The selection criteria used to define the study sample proved overly stringent and only a small number of subjects were able to participate. This limited the study power severely.

In addition to the deliberate bias designed into this study by sampling only households known to have relatively high levels of VOCs, a bias also exists in the manner in which households were selected to have well water sampled. Sampling by the IDPH was conducted at the request of residents of the study area. Therefore, most of the wells sampled belonged to people sufficiently concerned about VOC contamination to request the sampling; as a result, the group of wells sampled was not necessarily representative with respect to water levels of VOCs.

CONCLUSIONS

This study suggests air and water exposure to VOCs may result in elevated blood levels of these compounds. Air levels of 1,1-dichloroethylene were correlated with water levels, and inhalation exposures may contribute more to overall VOC body burden than do ingestion exposures. Limiting the sample to nonpregnant women aged 20 to 65 years who did not work outside the home more than 25 hours per week resulted in a small sample with minimal power.

RECOMMENDATIONS

Subsequent to the study USEPA made alternative sources of drinking water available to the residents of the study area. Since air levels of 1,1-dichloroethylene have been found to be correlated with water levels, the need for filters to eliminate VOCs from water used for purposes other than ingestion should be assessed. One possible approach would be to resample the 10 subjects now using bottled water and to compare blood levels prior to the use of bottled water with mean blood levels during the use of bottled water. Second, a questionnaire could be designed to collect detailed information on water use and other factors necessary to predict air levels of VOCs from water levels. Further blood sampling and interviewing may make it possible to assess the importance of the inhalation of volatilized VOCs from contaminated water on blood levels. Members of the community of southeast Rockford are

also participating in ATSDR's Trichloroethylene (TCE) Exposure Registry and are being contacted at yearly intervals concerning their health status.

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TABLES AND FIGURES

Household No. __ __ __ __
Interviewer number __ __
Date of interview __ __/__ __/__ __
Time of interview __ __ __ __
(Military time)

EPA NPL Site Number _ _ _ _ _ _ _ _ _ _ _ _ _

A. GENERAL INFO -- HOUSEHOLD

1. What is the actual address of the household?

   Street_____Apt._____
   City_____State_____
   Zip code_____

2. What is the mailing address of the household?

   Street_____Apt._____
   City_____State_____
   Zip code_____

3. In whose name is the household?

   _____

4. What is the telephone number of this household?

   (__ __ __) - __ __ __ - __ __ __ __

5. How many people live in this household?

   ___ ___

6. What was the previous address of this household?

   Street_____Apt._____
   City_____State_____
   Zip code_____

7. Please provide a name and address of someone outside your household who will know how to contact you if you move.

   Name_____
   Street_____Apt._____
   City_____State_____
   Zip code_____

Household No. __ __ __ __
Person number __ __ __ __

A. GENERAL INFO -- HOUSEHOLD (CONT.)

8. Please list all the people who live here with the information requested for each one. Start with the oldest member and proceed downward in age.

PERSON NO.	NAME	Sex	DATE OF BIRTH	DATE MOVED THIS ADDRESS	EDUCATION	OCCUPATION
01	_____	M=1	MO.__ __	MO.__ __	HIGHEST	_____
	_____	F=2	DAY__ __		GRADE	_____
	_____		YR.__ __	YR.__ __	COMPLETED:	_____
			AGE__ __		__ __	

PERSON NO.	NAME	Sex	DATE OF BIRTH	DATE MOVED THIS ADDRESS	EDUCATION	OCCUPATION
02	_____	M=1	MO.__ __	MO.__ __	HIGHEST	_____
	_____	F=2	DAY__ __		GRADE	_____
	_____		YR.__ __	YR.__ __	COMPLETED:	_____
			AGE__ __		__ __	

PERSON NO.	NAME	Sex	DATE OF BIRTH	DATE MOVED THIS ADDRESS	EDUCATION	OCCUPATION
03	_____	M=1	MO.__ __	MO.__ __	HIGHEST	_____
	_____	F=2	DAY__ __		GRADE	_____
	_____		YR.__ __	YR.__ __	COMPLETED:	_____
			AGE__ __		__ __	

PERSON NO.	NAME	Sex	DATE OF BIRTH	DATE MOVED THIS ADDRESS	EDUCATION	OCCUPATION
04	_____	M=1	MO.__ __	MO.__ __	HIGHEST	_____
	_____	F=2	DAY__ __		GRADE	_____
	_____		YR.__ __	YR.__ __	COMPLETED:	_____
			AGE__ __		__ __	

PERSON NO.	NAME	Sex	DATE OF BIRTH	DATE MOVED THIS ADDRESS	EDUCATION	OCCUPATION
05	_____	M=1	MO.__ __	MO.__ __	HIGHEST	_____
	_____	F=2	DAY__ __		GRADE	_____
	_____		YR.__ __	YR.__ __	COMPLETED:	_____
			AGE__ __		__ __	

Household No. __ __ __ __
Person number __ __ __ __

A. GENERAL INFO -- HOUSEHOLD (CONT.)

PERSON NO.	NAME	Sex	DATE OF BIRTH	DATE MOVED THIS ADDRESS	EDUCATION	OCCUPATION
06	_____	M=1	MO.__ __	MO.__ __	HIGHEST	_____
	_____	F=2	DAY__ __		GRADE	_____
	_____		YR.__ __	YR.__ __	COMPLETED:	_____
			AGE__ __		__ __	

PERSON NO.	NAME	Sex	DATE OF BIRTH	DATE MOVED THIS ADDRESS	EDUCATION	OCCUPATION
07	_____	M=1	MO.__ __	MO.__ __	HIGHEST	_____
	_____	F=2	DAY__ __		GRADE	_____
	_____		YR.__ __	YR.__ __	COMPLETED:	_____
			AGE__ __		__ __	

PERSON NO.	NAME	Sex	DATE OF BIRTH	DATE MOVED THIS ADDRESS	EDUCATION	OCCUPATION
08	_____	M=1	MO.__ __	MO.__ __	HIGHEST	_____
	_____	F=2	DAY__ __		GRADE	_____
	_____		YR.__ __	YR.__ __	COMPLETED:	_____
			AGE__ __		__ __	

PERSON NO.	NAME	Sex	DATE OF BIRTH	DATE MOVED THIS ADDRESS	EDUCATION	OCCUPATION
09	_____	M=1	MO.__ __	MO.__ __	HIGHEST	_____
	_____	F=2	DAY__ __		GRADE	_____
	_____		YR.__ __	YR.__ __	COMPLETED:	_____
			AGE__ __		__ __	

PERSON NO.	NAME	Sex	DATE OF BIRTH	DATE MOVED THIS ADDRESS	EDUCATION	OCCUPATION
10	_____	M=1	MO.__ __	MO.__ __	HIGHEST	_____
	_____	F=2	DAY__ __		GRADE	_____
	_____		YR.__ __	YR.__ __	COMPLETED:	_____
			AGE__ __		__ __	

[INTERVIEWER: WRITE THE PERSON NUMBER OF THE INDIVIDUAL ANSWERING THIS PART OF THE QUESTIONNAIRE: ___ ___]

Household No. __ __ __ __
Person number __ __ __ __

9. Where is your well located?
   1 = in the basement
   2 = outside of the house
   3 = other
   9 = don't know

10. Is your well open or sealed?
   1 = open
   2 = sealed
   9 = don't know

11. I'd like to ask you a question about the use of water filters. Does your home have a water filter?
   1 = yes
   2 = no (GO TO 12)
   9 = don't know (GO TO 12)

   11a. If yes, when was it installed? __ __/__ __
   MM YY

   11b. What was the brand name of the water filter? _____

   11c. What type of filter is it?
   1 = water softener (GO TO 11d)
   2 = water purifier
   3 = other (specify) (GO TO 11d) _____
   4 = combination (specify) _____
   9 = don't know (GO TO 11d)

   11c1. If water purifier, what type is it?
   1 = reverse osmosis
   2 = activated carbon
   3 = other (specify) _____
   9 = don't know

   11d. Where is the filter located?
   1 = tap
   2 = other location inside house
   3 = outside house
   9 = don't know

   11e. Is your filter serviced regularly?
   1 = yes
   2 = no
   9 = don't know

   11e1. If yes, how many times per year? __ __

Household No. __ __ __ __
Person number __ __ __ __

12. Does your household have a dishwasher?
    1 = yes
    2 = no
    9 = don't know

    12a. If yes, how many times per week do you use it? __ __

13. How many minutes per day does your household spend washing or rinsing dishes? __ __

14. Do you have a clothes washer?
    1 = yes
    2 = no (GO TO 15)
    9 = don't know (GO TO 15)

    14a. About how many loads of laundry do you wash per week? __ __

    14b. What water temperature setting does your household usually use on your washer?
    1 = hot
    2 = warm
    3 = cold
    9 = don't know

15. Do you have a dryer?
    1 = yes
    2 = no (GO TO 16)
    9 = don't know (GO TO 16)

    15a. About how many loads of laundry do you dry per week? __ __

    15b. Does the dryer have a vent to the outside of the house?
    1 = yes
    2 = no
    9 = don't know

16. Do you do any laundry by hand?
    1 = yes
    2 = no
    9 = don't know

    16a. About how many times per month is laundry done by hand? __ __

17. Do you have a whole house humidifier?
    1 = yes
    2 = no
    9 = don't know

    17a. If yes, how many hours per week has it been in use since June 1988?
    __ __

Household No. __ __ __ __
Person number __ __ __ __

18. Do you use a room humidifier in any room in the house?
    1 = yes
    2 = no (GO TO 19)
    9 = don't know (GO TO 19)

    18a. How many room humidifiers so you have? ___ ___

    18b. What type of humidifier is it?
         1 = warm mist
         2 = cool mist
         9 = don't know

19. Is there an open sump in your house?
    1 = yes
    2 = no
    9 = don't know

20. Do you have any leaks through cracks in your basement ?
    1 = yes
    2 = no
    9 = don't know

21. Does water collect in your basement?
    1 = yes
    2 = no
    9 = don't know

22. Do you have any untrapped drains?
    1 = yes
    2 = no
    9 = don't know

Household No. __ __ __ __
Person number __ __ __ __

Household No. __ __ __ __
Person No. __ __

The following questions concern your water consumption at home. A cup or glass is 8 ounces. We are interested in the period from June 1988 to the present.

23. Regarding unheated tap water which includes water directly from the faucet, or drinks made with unheated tap water such as orange juice or other juice, tea, koolaid, or any other beverage; on the average, how many full glasses or cups do you drink per day?

__ __ Full cups or glasses

23a. Do you add ice made from tap water to your unheated drinks?
1 = yes
2 = no
9 = don't know

23a1. If yes, how many cubes per glass? __ __

24. Including tea, coffee, cocoa, or any other hot beverage, on average, how many glasses or cups of hot beverages made from tap water do you drink per day?
__ __ Full cups or glasses

25. How many glasses or cups of heated or unheated bottled water or drinks made from bottled water do you drink per day?
__ __ Full cups or glasses

25a. Where do you get your bottled water? _____

26. How many glasses or cups of soda do you drink per day ?
__ __ Full cups or glasses

27. About how many cups of tap water would you estimate is used per day in cooking for any purpose such as making soup, cooking vegetables, etc., by any one in your household? __ __

Household No. __ __ __ __
Person number __ __ __ __

THE FOLLOWING QUESTIONS CONCERN SHOWERING OR BATHING. SIMILAR QUESTIONS WILL BE ASKED BY THE AIR SAMPLING PERSONNEL

28. How many times per week do you take a shower? __ __

    28a. How many minutes do you usually spend in the shower? __ __

29. How many times per week do you take a bath? __ __

    29a. How many minutes do you usually spend in the bath? __ __

30. What is the usual temperature of your shower or bath water?
    1 = very hot
    2 = moderately hot
    3 = warm
    4 = cool
    5 = cold
    9 = don't know

31. How many minutes do you usually spend in the bathroom after taking a shower or bath? __ __

32. Do you shower or bathe with the bathroom door open or shut?
    1 = open
    2 = shut
    9 = don't know

33. Do you use an exhaust fan in your bathroom when taking a bath or shower?
    1 = yes
    2 = no
    9 = don't know

34. When you are in the bathroom after taking a shower or bath, is there steam in the bathroom?
    1 = yes
    2 = no
    9 = don't know

35. At home, do you help anyone bathe or take a shower, such as a child or an elderly person?
    1 = yes
    2 = no (GO TO 36)
    9 = don't know (GO TO 36)

    35a. If yes, on the average, how many times per week do you assist in the bathing or showering? __ __

    35b. On the average, how many minutes do you usually spend with them in the bathroom each time? __ __

36. How many times per day do you wash your hands at home? __ __

Household No. __ __ __ __
Person number __ __ __ __

37. Do you wash a car or other vehicle at home?
    1 = yes
    2 = no
    9 = don't know

    37a. If yes, how often do you wash it?
        1 = once per week
        2 = twice per month (every other week)
        3 = once per month
        4 =less than once per month
        9 = don't know

38. Do you wash your pets at home?
    1 = yes
    2 = no
    9 = don't know

    38a. If yes, how many times do you wash them?
        1 = once per week
        2 = twice per month (every other week)
        3 = once per month
        4 =less than once per month
        9 = don't know

    38b. How many minutes at a time? __ __

Household No. __ __ __ __
Person number __ __ __ __

THE NEXT QUESTIONS CONCERN ACTIVITIES THAT YOU MAY HAVE BEEN INVOLVED IN DURING THE PAST FEW DAYS.

39. Have you been to the dry cleaners in the past 3 days?
1 = yes
2 = no
9 = don't know

39a. If yes, how many days ago were you there? __

40. Have you pumped your own gasoline in the past 3 days?
1 = yes
2 = no
9 = don't know

40a. If yes, how many days ago did you pump gasoline? __

41. Do you presently drink alcoholic beverages?
(1 DRINK = 1 BEER, 1 SHOT LIQUOR, OR 1 GLASS WINE)
1 = yes
2 = no
41a. On the average in the past 6 months, how many drinks a week do/did you have? ___ ___

42. Have you smoked at least 100 cigarettes during your entire life?
(1 PACK = 20 CIGARETTES)
1 = yes
2 = no (GO TO 43)

42a. Do you smoke cigarettes now?
1 = yes
2 = no (GO TO 43)

42a1. On the average, how many cigarettes a day do you now smoke?
___ ___

42a2. When did you smoke your last cigarette? __ __ __

43. Have you ever used any other tobacco products such as cigars, pipe, chewing tobacco, or snuff?
1 = cigars/pipe
2 = chewing tobacco
3 = snuff
4 = combination of above
5 = none of the above
9 = don't know

Household No. __ __ __ __
Person number __ __ __ __

44. Have you done any house painting in the past month?
1 = yes
2 = no
9 = don't know

44a. If yes, when did you do the painting? __ __
no. of days

45. Have you done any remodelling other than house painting in the past month?
1 = yes
2 = no
9 = don't know

45a. If yes, when did you do the remodelling? __ __
no. of days

45b. What type of remodelling was it? _____

46. Does anyone in the household have any of the following hobbies?
1 = furniture refinishing
2 = painting
3 = automobile repair
4 = metal working
5 = jewelry making
6 = combination of above
9 = don't know

47. Does anyone in your household work as a
1 = dry cleaner
2 = mechanic or gas station attendant
3 = metal worker
6 = combination of above
8 = other occupation with solvent exposure (specify _____)
9 = don't know
10 = none of the above

47a. If yes to any of the above, does this person wear his (her) workclothes home?
1 = yes
2 = no
9 = don't know

47b. If yes to any of the above, does this person launder his (her) work clothes at home?
1 = yes
2 = no
9 = don't know

THESE ARE ALL THE QUESTIONS I HAVE. DO YOU HAVE ANY QUESTIONS? THANK YOU FOR YOUR TIME.

AIR SAMPLING FORM

Part A (to be completed by interviewer)

1) Household No. _____

2) Interviewer No. _____

3) Date of Interview ___/___/___

4) Time of interview ___ ___ ___ ___
(military time)

(Detach form and leave with participant)

Part B (to be completed by air sampler)

1) Date of Sampling ___/___/___

2) How long since the shower/tub was last used (in hours)?

a) shower _____

b) bathtub _____

3) In the past 24 hours, how many showers/baths were taken?

a) shower _____

b) bath _____

4) What is the average amount of time for a shower/bath (in minutes)?

a) shower _____

b) bath _____

5) How long since the washer/dryer was last used (in hours)? (if no washer and dryer, go to question 8)

a) washer _____

b) dryer _____

c) no washer

d) no dryer

6) How many times was the washer/dryer used in the last 24 hours?

a) washer _____

b) dryer _____

7) What is the average cycle time for the washer/dryer (in minutes)?

a) washer _____

b) dryer _____

8) How long since the dishwasher was last used (in hours)? _____ (if no dishwasher, go to question 11)

9) How many times in the past 24 hours was the dishwasher used? _____

10) What is the average cycle time for the dishwasher (in minutes)? _____

11) How long since the sink was used to wash dishes (in hours)? _____

12) How many sinkfuls of dishes were washed in the past 24 hours? _____

13) How long does it take on average to wash a sinkful of dishes (in minutes)? _____

14) Have you run a humidifer in the past 24 hours?

a) No

b) Yes (if yes, for how long _____ minutes)

15) How much water was used in the last 24 hours for cooking or brewing (in cups)? _____

16) What other indoor uses of the household water have taken place in the last 24 hours (e.g., mopping floors, shampooing rugs, washing pets, etc.)? _____

_____

17) Air Bag Sampling (shower)

a) Location _____

b) No. of Samples _____

c) Type of bag _____

d) Sample Identifiers _____

_____

e) How long was shower run? _____

f) Description of collection _____

_____

_____

18) Tenax/CMS Samples (shower)

a) Time Started _____ Time ended _____

b) Pump No. _____ Flow rate _____

_____ _____

_____ _____

c) Calibration _____

_____

d) No. of Samples _____

e) Sample Identifiers _____

f) How long was shower run? _____

g) Description of collection _____

_____

_____

19) Tenax/CMS Samples (household)

a) Time Started (indoor) _____ Time ended _____

Time Started (outdoor) _____ Time ended _____

b) Pump No. _____ Flow Rate _____

_____ _____

_____ _____

_____ _____

c) Calibration _____

_____

d) No. of Samples _____

e) Sample Identifiers _____

f) Description of Collection _____

_____

_____

20) Tenax/CMS Samples (laundry)

a) Time started _____ Time ended _____

b) Pump No. _____ Flow Rate _____

_____ _____

_____ _____

_____ _____

c) Calibration _____

_____

d) No. of Samples _____

e) Sample Identifiers _____

f) Description of Collection _____

_____

_____

21. Comments _____

_____

_____

_____

_____

Sampler Initials _____

APPENDIX 2
Analysis Protocols

PROTOCOL FOR MEASUREMENT OF VOLATILE ORGANIC COMPOUNDS IN HUMAN BLOOD USING PURGE/TRAP GAS CHROMATOGRAPHY MASS SPECTROMETRY

Toxicology Branch
Division of Environmental Health Laboratory Sciences
Center for Environmental Health and Injury Control
Centers for Disease Control
Public Health Service
U.S. Department of Health and Human Services

Introduction

This protocol describes methods developed and used at the Centers for Disease Control for the measurement of volatile organic compounds (VOCs) in human blood. This is a purge and trap (direct sparging with helium) gas chromatographic method using high resolution mass spectrometric detection in the full scan mode. The method is applicable to the determination of the 35 following compounds in 10 mL blood at approximately the detection limits given.

Analyte	Detection Limit (ppb)
1,1,1-Trichloroethane	0.04
1,1,2,2-Tetrachloroethane	0.01
1,1,2-Trichloroethane	0.02
1,1-Dichloroethane	0.01
1,1-Dichloroethene	0.02
1,2-Dichlorobenzene	0.03
1,2-Dichloroethane	0.01
1,2-Dichloropropane	0.01
1,3-Dichlorobenzene	0.04
1,4-Dichlorobenzene	0.03
2-Butanone	0.3
Acetone	200
Benzene	0.03
Bromodichloromethane	0.01
Bromoform	0.02
Carbon Disulfide	2
Carbon Tetrachloride	0.02
Chlorobenzene	0.01
Chloroform	0.02
cis-1,2-Dichloroethene	0.01
Dibromochloromethane	0.02
Dibromomethane	0.02
Ethylbenzene	0.02
Hexachloroethane	0.1

Hexane	2
m-/p-Xylene	0.02
Methylene chloride	0.05
o-Xylene	0.03
Styrene	0.01
Tetrachloroethene	0.02
Toluene	0.1
trans-1,2-Dichloroethene	0.02
Trichloroethene	0.005

Quantitation is achieved by isotope dilution in all cases by reference to commercially available labelled isotopes.

Summary

VOCs in whole blood are determined by heated purge and trap gas chromatography mass spectroscopy (GC/MS). Stable isotopically labelled analogs of the compounds of interest are added to 10 mL blood and this entire sample is injected into a specially designed sparging vessel which is already attached to the purging apparatus via air-tight seals. Prepurified helium gas is bubbled through the blood which is heated to approximately 35°C. This process removes volatile compounds from the sample into the gas stream. The purged volatile compounds pass into and are captured by a Tenax trap. Once the 15 minute purge cycle is complete, the Tenax trap is purged with dry helium gas for 6 minutes to remove absorbed water. The trap is then heated to 180°C for 4 minutes to desorb all volatile compounds. As the compounds are desorbed, they are trapped at the gas chromatograph injection port by a liquid nitrogen trap at -150°C. Following this period, the site is ballistically heated to 200°C injecting the compounds onto the DB-624 capillary column which is interfaced to the mass spectrometer. The mass spectrometer is operated in the full scan mode (40 - 200 amu) with one scan collected per second. Quantitation is accomplished from specific ion responses relative to those of the corresponding labelled analogs. The responses of analytes and analogs are corrected for contributions from each other through the use of an isotope dilution calculation. Final determinations are made based on six-point calibration curves and the concentrations are normalized according to sample weight.

Interferences

Compounds with similar chromatographic properties and characteristic mass spectral ions as the compounds of interest may interfere with the analysis. Care must be exercized in determining possible sources of these interferences and in some cases alternate ions should be selected to eliminate these. The use of high resolution mass spectrometry has proven to be of immense aid in this respect and has been shown to be absolutely necessary in certain cases.

Interferences which have their source in the measurement apparatus itself should be examined by determination of instrument blanks. For this purpose, a pure water sample remains attached to the measuring apparatus and is examined regularly to check for operational levels of instrument blanks. By leaving this sample attached to the measurement apparatus, exposure to airborne contaminants is eliminated and the level of volatiles in the sample are reduced to a

nondetectable level.

Glassware used for standards must be examined for sources of contamination. All glassware is heated in a vacuum oven at 150°C for at least 8 hours to remove adsorbed volatiles. The vacuum oven used contains an independent vacuum source since cross-contamination from other laboratory operations has been determined to be a major source of contamination of laboratory glassware. This glassware is cooled to room temperature before removal from the oven and sealed to diminish exposure to volatile compounds which are present in laboratory air.

The water used for dilution of standards and as water blanks is an extremely critical potential source of contamination. Numerous sources of water were examined to determine the most volatile-free water available. No commercial filtering or purification system was found which could consistently yield water at acceptable levels (< 20 ppt for most analytes). An acceptable source of water was discovered at a non-commercial site and all further studies make use of this source. Under some circumstances this source of water fails to yield acceptable levels of volatile organic compounds. In this case, the water is further purified to yield blank water with acceptable levels of VOCs. To prevent further contamination from the laboratory air, water samples are sealed in glass ampules. To provide consistency in measurement, all water samples used for a specific calibration curve have the same origin and are sealed at the same time. In all cases typical blank water levels are below the detection limits given above.

Since all commercially available vacutainers contain measureable levels of VOCs, the lots used to collect blood samples must be examined to determine levels of contamination present. This is accomplished by adding well characterized blank water to the vacutainers, allowing a reasonable exposure time to both the tube and the stopper and then characterizing these specimens for concentration of VOCs. In spite of these efforts, some compounds still have substantial contamination levels when compared to background levels of these compounds in blood. In particular, methylene chloride, dibromomethane, and carbon disulfide have large levels of interfering contamination in untreated vacutainers. Other analytes, including bromoform, chloroform, hexane, etc., also show contamination from vacutainers to a varying extent. Further efforts have been made to remove contamination from the vacutainers. This involves disassembly, heating in a vacuum oven, reassembly, restoration of vacuum, and sterilization. This process removes a substantial fraction of interfering analytes.

Contamination by carryover must be examined over the entire range of analyte concentrations expected. The current purge and trap apparatus did not show any appreciable carryover for the analytes being measured over the standard concentration range presented here.

Safety

Many of the compounds used in this study are considered to be toxic or carcinogenic hazards. They should be treated as a potential health hazard in all cases. Always work under a chemical fume hood when transferring these materials. Use a high draft fumehood and lower

all the sashes because a number of these compounds are strong lachrymators and cause severe eye irritation at low concentrations. Wear appropriate gloves when handling these chemicals because all of them are readily absorbed through the skin.

Analyte concentrations in blood samples are at trace levels and therefore due not pose a substantial chemical hazard to personnel. Even though there is a minimal chemical hazard due to these samples, the microbiological hazard associated with whole blood samples necessitates the treatment of all blood samples as potential health hazards. Biosafety level 2 procedures should be followed when handling blood samples. These procedures include handling blood samples with protective gloves within a biological safety cabinet. After the sample has been analyzed it should be decontaminated with a chemical disinfectant and disposed as chemical waste. All glassware, etc. that contacts the blood samples should be treated as contaminated and autoclaved before disposal.

Standard Preparation

Positive displacement pipets are used for all transfer of liquids in the uL range. Transfers in the 5-30 uL range use a pipet with 0.1 uL increments. Transfers in the 31-100 uL range use a pipet with 0.2 uL increments. Transfers in the 101-250 uL range use a pipet with 1 uL increments. 25 mL class A sealable volumetric flasks are used to make all standards. Weights of neat compounds are determined on an analytical balance to the nearest 0.1 mg.

Standards are prepared with the following final concentrations (ppb):

Compound	# 1	# 2	# 3	# 4	# 5	# 6
1,1,1-Trichloroethane	0.05	0.1	0.5	1	5	10
1,1,2,2-Tetrachloroethane	0.005	0.01	0.05	0.1	0.5	1
1,1,2-Trichloroethane	0.007	0.014	0.07	0.14	0.7	1.4
1,1-Dichloroethane	0.005	0.01	0.05	0.1	0.5	1
1,1-Dichloroethene	0.01	0.02	0.1	0.2	1	2
1,2-Dichlorobenzene	0.005	0.01	0.05	0.1	0.5	1
1,2-Dichloroethane	0.004	0.009	0.04	0.09	0.4	0.9
1,2-Dichloropropane	0.005	0.01	0.05	0.1	0.5	1
1,3-Dichlorobenzene	0.004	0.009	0.04	0.09	0.4	0.9
1,4-Dichlorobenzene	0.04	0.08	0.41	0.8	4	8
2-Butanone	0.06	0.12	1.2	2.5	12	25
Acetone	200	400	900	1800	3800	6000
Benzene	0.01	0.02	0.1	0.2	1.2	2
Bromodichloromethane	0.005	0.01	0.05	0.1	0.5	1
Bromoform	0.02	0.04	0.20	0.4	2	4
Carbon Disulfide	2.4	4.8	20	40	80	120
Carbon Tetrachloride	0.004	0.009	0.04	0.09	0.4	0.9
Chlorobenzene	0.004	0.009	0.04	0.09	0.4	0.9

Chloroform	0.009	0.018	0.09	0.18	0.9	1.8
cis-1,2-Dichloroethene	0.009	0.018	0.09	0.18	0.9	1.8
Dibromochloromethane	0.004	0.009	0.04	0.09	0.4	0.9
Dibromomethane	0.02	0.04	0.2	0.4	2	4
Ethylbenzene	0.01	0.02	0.1	0.2	1	2
Hexachloroethane	0.006	0.012	0.06	0.12	0.6	1.2
Hexane	4	8	32	64	130	190
m-Xylene	0.01	0.02	0.20	0.41	2.1	4.1
Methylene chloride	0.02	0.04	0.42	0.83	4.2	8.3
o-Xylene	0.025	0.05	0.25	0.5	2.5	5
p-Xylene	0.01	0.02	0.20	0.41	2.1	4.1
Styrene	0.008	0.017	0.08	0.17	0.8	1.7
Tetrachloroethene	0.02	0.04	0.2	0.4	2	4
Toluene	0.01	0.02	0.2	0.4	2	4
trans-1,2-Dichloroethene	0.01	0.02	0.1	0.2	1	2
Trichloroethene	0.004	0.009	0.04	0.09	0.4	0.9

Native analyte standards are made by succesive dilution in methanol from the neat compounds. Because of variation in the volatility of these compounds, the use of concentrated stock solutions for long term storage is unacceptable. The intermediate stock solutions are prepared fresh from the neat compounds every three months. These solutions are sealed in glass ampules and placed in a -60°C freezer until used. This has proven to be a successful method of preserving standard integrity. On the day of use, the standard is prepared by dilution of the appropriate volume of these intermediate stock solutions into 25 mL of 'contaminant-free' water.

Labelled analog solutions are prepared to achieve the following approximate final concentrations (ppb):

1,1,1-Trichloroethane-D_3	1
1,1,2,2-Tetrachloroethane-D_2	0.1
1,1,2-Trichloroethane-D_3	0.1
1,1-Dichloroethane-D_3	0.1
1,1-Dichloroethene-D_2	0.2
1,2-Dichlorobenzene-D_4	0.25
1,2-Dichloroethane-D_4	0.2
1,2-Dichloroethene-D_2 (mix)	0.2
1,2-Dichloropropane-D_6	0.2
1,4-Dichlorobenzene-D_4	2
2-Butanone-D_3	3
Acetone-$^{13}C_3$	25
Benzene-$^{13}C_6$	0.2
Bromodichloromethane-^{13}C	0.1
Bromoform-^{13}C	0.5
Carbon disulfide-^{13}C	5

Carbon tetrachloride-^{13}C	0.1
Chlorobenzene-D_5	0.2
Chloroform-^{13}C	0.1
Dibromochloromethane-^{13}C	0.1
Dibromomethane-^{13}C	0.1
Ethylbenzene-D_{10}	0.4
Hexachloroethane-1-^{13}C	0.5
Hexane-D_{14}	4
Methylene chloride-^{13}C	0.5
o-Xylene-$^{13}C_2$	0.2
p-Xylene-D_{10}	0.7
Styrene-D_8	0.2
Tetrachloroethene-^{13}C	0.1
Toluene-D_8	0.2
Trichloroethene-^{13}C	0.05

Labelled analog solutions are made by succesive dilution in methanol from the neat compounds. Concentrated stock solutions are stored in sealed ampules at -60°C. Because of the major expense in acquiring these label analogs further dilutions are made from these concentrated stock solutions. Intermediate stock solutions of these analogs are prepared fresh every three months. These solutions are sealed in glass ampules and stored at -60°C until used. On the day of use, the analog solution is prepared by dilution of the appropriate volume of intermediate stock solution into 25 mL of methanol. 20 uL of this analog solution is added to each blank, standard, whole blood, or quality control sample before injection into the purging apparatus.

Sample Collection

Previous studies of VOCs indicate that their half-life in human blood is relatively short. In most cases, values between 6 and 24 hours are considered to be the best estimates for these half-lives. Because VOCs do not reside long in the body, special sample collection considerations are necessary. Except in cases of extremely high exposure, sampling of blood after as much as 3 days after removal from exposure will not indicate abnormal levels in the blood. Of course the length of time after exposure for which useful samples can still be obtained will vary with the level of exposure. It is therefore suggested that samples be obtained either before removal from exposure or as quickly after this time as possible. This will require preparation well before assessment of environmental levels. Thus, it is highly desirable that protocols, release forms, and sample collection materials be ready and on hand when assessment begins. This will enable the collection of blood samples before the VOCs are excreted from the body.

Samples are collected by venipuncture using grey top vacutainers which contain potassium oxalate / sodium fluoride as anticoagulant. Two 10 mL tubes are collected from each individual, the second tube being used for examination of reproducibility and sample stability. Within 15 minutes the samples are placed on wet ice or stored at refrigerator temperatures. The

samples should not be frozen or allowed to stand for an extended length of time at room temperature.

Samples should be shipped via next day carrier in insulated containers along with enough ice packs so that the temperature can be maintained during the shipping process. Shipments should not be made which will arrive on weekends or federal holidays. All shipments are made to

Centers for Disease Control
Bldg 17, Room 1814, F17
4770 Buford Highway
Chamblee, Georgia 30341
Attn: Dr. David Ashley

Preliminary experiments have indicated that the concentration of some volatile analytes changes over sample storage time. Therefore, the samples should be shipped within 1 - 2 days of collection so that they can be analyzed within 2 - 3 weeks of collection.

Sample Preparation

Before sample introduction the purging vessels are thoroughly cleaned with methanol, heated overnight, a small portion of antifoam agent added, and the vessels heated and purged through one regular purge and trap cycle with no sample present. This assures the removal of any remaining contamination from the vessel and antifoam agent.

10 mL samples are extracted into a gas-tight syringe which has been thoroughly washed with methanol and 'contaminant-free' water. The syringe is weighed to the nearest 0.01 g both before and after extraction of the sample. These numbers are subtracted to determine the sample weight. To the syringe is added 20 uL of the analog solution. The syringe is attached to the purging apparatus via a luerlock fitting, the valve is opened, the sample is injected into the purging vessel, and the valve is closed before removal of the syringe. A typical daily work load consists of an instrument blank, a water blank, an analytical standard, 3 unknowns, and a quality control sample.

Instrumentation

The purge and trap apparatus consists of a Tekmar LSC 2000 purge and trap concentrator with an attached ALS 2016 automated sampler. This system allows up to 16 samples to be loaded for processing at one time. Because of lack of communication between the sampler and the mass spectrometer data system, the method requires operator attention at particular steps in the analysis routine. Helium flow rate is maintained at 30 ml/min at 20 psi. This flow rate is critical since too large a flow will cause column breakthrough and reduce sensitivity to low boiling compounds. For sample analysis the following steps are programmed into the purge and trap concentrator.

Step	Time	Temperature
Preheat	3.00 min.	30°C
Purge	15.00 min.	30°C
Dry Purge	6.00 min.	30°C
Cap Cooldown		-150°C
Desorb Preheat		175°C
Desorb	4.00 min.	180°C
Inject	0.75 min.	200°C
Bake	36.00 min.	225°C

Analyte separation is carried out on a Hewlett-Packard model 5890 gas chromatograph specifically modified to allow channeling of effluent through a heated interface into the mass spectrometer. The chromatograph is equipped with a J & W 30m DB-624 column with 1.8 um film thickness. The gas chromatograph uses the following temperature program:

Temperature	Hold Time	Rate (°/min.)	Ramp Time
0°C	1.5 min.	12.0	2.5 min.
30°C	2.0 min.	8.0	20.0 min.
190°C	10.0 min.		

The mass spectrometer is a VG Analytical 70E high resolution mass spectrometer operating at 3000 resolving power. Instrument tuning and resolution must be checked before each experimental run. Masses are calibrated versus perfluorokerosene. The instrument is operated in full scan mode (40 - 200 amu).

The following masses are used as quantitation ions:

Analyte	Quantitation Ion	Analog	Quantitation Ion
1,1,1-Trichloroethane	99.9800	1,1,1-Trichloroethane-D_3	96.9612
1,1,2,2-Tetrachloroethane	82.9455	1,1,2,2-Tetrachloroethane-D_2	83.9518
1,1,2-Trichloroethane	96.9612	1,1,2-Trichloroethane-D_3	99.9800
1,1-Dichloroethane	63.0001	1,1-Dichloroethane-D_3	66.0189
1,1-Dichloroethene	95.9534	1,1-Dichloroethene-D_2	64.9941
1,2-Dichlorobenzene	145.9690	1,2-Dichlorobenzene-D_4	151.9912
1,2-Dichloroethane	61.9923	1,2-Dichloroethane-D_4	67.0060
1,2-Dichloropropane	63.0002	1,2-Dichloropropane-D_6	67.0253
1,3-Dichlorobenzene	145.9690	1,4-Dichlorobenzene-D_4	151.9912
1,4-Dichlorobenzene	145.9690	1,4-Dichlorobenzene-D_4	151.9912
2-Butanone	72.0575	2-Butanone-D_3	75.0764
Acetone	59.0452	Acetone-$^{13}C_3$	61.0519
	60.0486		
Benzene	78.0470	Benzene-$^{13}C_6$	84.0671
	77.0391		
Bromodichloromethane	82.9455	Bromoform-^{13}C	173.8458

Bromoform	172.8425	Bromoform-^{13}C	173.8458
Carbon Disulfide	77.9399	Carbon disulfide-^{13}C	78.9433
	75.9441		
	79.9357		
Carbon Tetrachloride	116.9066	Carbon tetrachloride-^{13}C	117.9099
Chlorobenzene	112.0080	Chlorobenzene-D_5	117.0394
Chloroform	82.9455	Chloroform-^{13}C	83.9489
cis-1,2-Dichloroethene	95.9534	cis-1,2-Dichloroethene-D_2	64.9941
Dibromochloromethane	128.8923	Chlorodibromomethane-^{13}C	129.8958
Dibromomethane	92.9339	Bromoform-^{13}C	173.8458
Ethylbenzene	106.0783	Ethylbenzene-D_{10}	116.1410
Hexachloroethane	165.8725	Hexachloroethane-1-^{13}C	166.8758
Hexane	86.1096	Hexane-D_{14}	100.1974
	87.1129		
m-/p-Xylene	106.0783	p-Xylene-D_{10}	116.1410
Methylene chloride	83.9534	Methylene chloride-^{13}C	84.9567
o-Xylene	106.0783	o-Xylene-$^{13}C_2$	116.1410
Styrene	104.0626	Styrene-D_8	112.1128
Tetrachloroethene	165.8725	Tetrachloroethene-^{13}C	166.8758
Toluene	91.0548	Toluene-D_8	98.0987
	65.0391		
trans-1,2-Dichloroethene	95.9534	trans-1,2-Dichloroethene-D_2	64.9941
Trichloroethene	129.9144	Trichloroethene-^{13}C	130.9177

In some cases, two or more masses are indicated. These allow a wider dynamic range in measurement for certain analytes.

Data Analysis

Data are processed automatically by a chromatogram generation, peak detection and quantitation routine specifically designed for this application. This routine provides for hard copy output of all chromatograms with peak detection indicated, full scan spectra at all detected peak maxima, and highly expanded mass spectra around the ions of interest. All peaks which are automatically detected are individually checked for proper integration. All spectra are checked for appearance of interferences which may occur in the quantitation windows. Correction is made for the o-xylene contribution to the styrene signal.

Isotope Dilution Calculations

Quantitation is achieved by determination of relative response factors between native analytes and labelled analogs added to the sample being examined. The analog levels are kept constant and calibration curves of the relative response between analyte and analog are plotted for standards at five different concentrations which cover the range of interest. In most cases, native analytes have some response at the mass used for determination of contribution for the

labelled isotope. Likewise, the labelled isotope often contributes some response at the mass being used for analyte quantitation. In these cases corrections must be made for the contribution of labelled analyte to the native ion and vice versa. In order to properly determine relative response factors between the analyte and analog, these effects must be taken into account. This is the basis for the use of more complex isotope dilution calculations.

The following ratios must be determined for correct use of these calculations.

For the native analytes,

$$Rx = \frac{\text{area of quantitation ion of the analyte at analyte retention time}}{\text{area of the analyte contribution to the quantitation ion of the labelled analog at analyte retention time}}$$

For the labelled analogs,

$$Ry = \frac{\text{area of labelled analog contribution to the quantitation ion of the analyte at analog retention time}}{\text{area of the quantitation ion of the labelled analog at analog retention time}}$$

These ratios are a measure of the degree to which the native analytes contribute labelled analog signal and vice versa. If measured correctly, Ry will also account for the presence of native analyte present in the labelled spiking solution. These ratios are measured by adding enough of the solutions of interest to overwhelm any contribution from contamination.

If no area is detected in the denominator of the Rx calculation, Rx should be set to a number substantially larger than 1. In this protocol 1000000 was chosen. If no area is detected in the numerator of the Ry calculation, Ry should be set to a number substantially smaller than 1. In this protocol 0.000001 was chosen. This will allow use of the same calculation in cases in which there is no contribution of analog to analyte signal or analog to analyte signal.

The ratio of the analyte signal to the analog signal must then be determined for each sample, standard, blank or QC material. This is given as

$$Rm = \frac{\text{area of the quantitation ion of the analyte at analyte retention time}}{\text{area of the quantitation ion of the labelled analog at analog retention time}}$$

The relative response is calculated from the above ratios as

$$RR = \frac{(Ry - Rm)(Rx + 1)}{(Rm - Rx)(Ry + 1)}$$

For this calculation to remain valid Rm must be between 2Ry and 0.5 Rx. Otherwise large deviations occur for small errors. For standards, the relative response is divided by the weight of the standard solution and this value is plotted versus standard concentration to create calibration curves. Slopes from these calibration curves are determined using linear regression analysis and used to determine unknown concentrations using:

$$Conc_{unknown} = \frac{(RR_{unknown}\text{-intercept})}{slope} \times \frac{1}{weight_{unknown}}$$

Data Transfer and Processing

Results files are transferred directly to the EHLS local area network where all quantitation and summary calculations are accomplished. Quantitation is achieved by comparison of calculated relative response factors to calibration curves generated from standards which are measured during the same period. When a new set of standards is introduced, the calibration curves are recalculated to account for any minor variation in analog concentration from one standard set to the next.

Quality Assurance/Quality Control

Quality control consists of daily experimental checks on the stability of the analytical system and standards and quality control materials which are added to each day's run sequence. A 'pure-water' blank sample is run at the beginning of each day to check for the presence of contamination in the purge and trap system or the labelled analog solution. In addition, determination of label ion counts for this material is used to check daily method sensitivity. Relative retention times are examined for each analyte to insure the choice of the correct chromatographic peak.

Serum quality control materials were developed from bulk serum spiked with the analytes being examined in this study. This material was sealed in ampules and frozen at -60°C to insure long term stability. Quality control materials were developed at two different analyte concentration levels. These samples have proven to be stable over the entire time that the study has been underway. Individual quality control charts are created for each analyte examined and quality control limits are used to insure analytical stability. 99% confidence limits are used as the first analytical control limits. Additional limits include rejection for seven consecutive analytical measurements either above or below the mean and rejection of seven values which show trends in either the positive or negative direction.

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Dear Resident:

The Illinois Department of Public Health is conducting a study with the assistance from the U.S. Public Health Service, Agency for Toxic Substances and Disease Registry (ATSDR) and the Winnebago County Health Department. The study is intended to determine the extent which residents are exposed to volatile organic compounds as a result of the ground water contamination in Southeast Rockford. Your household has been identified as one which may be included in our study.

We recently tried to contact you, but you were not at home. We would like to discuss this study and your possible participation in it. Please contact Dr. Jane Keller at 987-7511 so we can schedule a time to meet with you.

Illinois Department of Public Health
4302 North Main Street
Rockford, IL
815/987-7511

Participant consent: I have read the description of this survey. All of my questions have been satisfactorily answered. I voluntarily request that I be included in this survey. I understand and agree that a photostatic copy or fascimile of this consent will be as valid as the original even though such copy does not contain the original writing of my signature.

*The VOCs that will be measured are: 1, 1-dichloroethane, 1, 1-dichloroethylene, 1,2-dichloroethane, 1,2-dichloroethylene (cis and trans), 1,1,1-trichloroethane, trichloroethylene, and tetrachloroethylene.

Participant name (print)_____

Participant signature_____

Date _____ Witness _____

PARTICIPANT CONSENT

The Illinois Department of Public Health, with assistance from the Agency for Toxic Substances and Disease Registry of the Public Health Service, is conducting a study of possible exposure to volatile organic compounds* (VOCs) among selected residents of S.E. Rockford. My participation will help determine whether there is exposure to VOCs in my home.

The survey has three parts: a questionnaire, a blood test for exposure to VOCs and in-home air monitoring. My part in the survey will include:

1. Answering questions about habits and activities in my home and about the occupations of adults in my home.

2. Allowing testing on a blood sample of approximately 20 ml (two small test tubes) which will be taken with a needle from a vein in the arm. There is little risk associated with this procedure. Temporary discomfort and a small bruise may occur at the site where the needle enters the skin. I may be asked to provide a second sample at a future date to measure changes over time.

3. Allowing in-home air monitoring to test for VOCs in my home. A monitor will be set up in my home for approximately 24 hours. This monitoring will require the state health department or their representatives to enter my home, place an air monitor and then retrieve it 24 hours later.

Participation: I understand that my active participation will take about 30 minutes. There will be no physical examination. There is no provision for compensation or medical treatment in the event of injury as a result of my participation. I understand that I can stop my participation at any time. If I choose not to participate or to stop at any time there will be no penalty. Any benefits which I now receive or to which I am entitled will not be affected by this decision.

Results: As a result of my participation in this survey I will receive a blood test for VOCs at no charge. The Illinois Department of Public Health will send me a letter as soon as verified results are received and will refer me for a medical evaluation if it is indicated from my test results.

Confidentiality: I understand that the state health department will take every reasonable precaution to keep my records confidential. Any information shared with the Agency for Toxic Substances and Disease Registry or Centers for Disease Control will be kept in accordance with the Federal Privacy Act of 1974. Any reports of this survey will not identify specific individuals, and will only give group information.

ILLINOIS DEPARTMENT OF
PUBLIC HEALTH
A Healthier Today For A Better Tomorrow

Bernard J. Turnock, M.D., Director

October 18, 1989

Dear Rockford Resident:

The Illinois Department of Public Health, in conjunction with the U.S. Public Health Service, Agency for Toxic Substances and Disease Registry (ATSDR) and the Winnebago County Health Department, is conducting an important health study that will take place in your neighborhood during the next three weeks. The study will determine whether people potentially exposed to volatile organic compounds in groundwater have these chemicals in their blood.

While your participation is voluntary, we hope that, if eligible, you will choose to participate. The study will consist of three phases: 1) completing a questionnaire, which will take about 20 minutes, 2) blood sampling, which will take about 10 minutes, and 3) air sampling of a few participants' houses.

I hope we can count on your help with this important study. If you have any questions, please call Dr. Jane Keller at the Rockford regional office at 987-7511.

Sincerely;

Bernard J. Turnock, M.D.
Director, Public Health

Rockford Regional Office
4302 North Main Street • Rockford, Illinois 61103

APPENDIX 1

The contents of Appendices 1 and 2 are presented in their entirety as submitted by the authors for informational purposes and have not been revised or edited to conform with Agency for Toxic Substance and Disease Registry guidelines.

Table 11 - Exposure to confounding variable versus elevated blood levels of volatile organic compounds. Continued.

	Blood Levels Elevated	Blood Levels Not Elevated	Total
Hobbies			
Exposed	0	1	1
Not exposed	4	5	9
Total	4	6	10
Occupations Involving Solvent Exposure			
Exposed	2	5	7
Not exposed	2	1	3
Total	4	6	10
Workclothes Worn Home From Job With Potential Solvent Exposure			
Exposed	2	5	7
Not exposed	2	1	3
Total	4	6	10
Workclothes From Job With Potential Solvent Exposure Laundered at Home			
Exposed	1	4	5
Not exposed	3	2	5
Total	4	6	10
Exposure to Any of the Above Confounders			
Exposed	3	6	9
Not exposed	1	0	1
Total	4	6	10

Table 11 - Exposure to confounding variable versus elevated blood levels of volatile organic compounds.

	Blood Levels Elevated	Blood Levels Not Elevated	Total
Dry Cleaner			
Exposed	0	0	0
Not exposed	4	6	10
Total	4	6	10
Pumping Gasoline			
Exposed	0	1	1
Not exposed	4	5	9
Total	4	6	10
Present Alcohol Consumption			
Exposed	0	3	3
Not exposed	4	3	7
Total	4	6	10
Current Smoking			
Exposed	1	2	3
Not exposed	3	4	7
Total	4	6	10
Housepainting			
Exposed	0	1	1
Not exposed	4	5	9
Total	4	6	10
Remodeling			
Exposed	1	2	3
Not exposed	3	4	7
Total	4	6	10

Table 10 - Comparison of mean levels of volatile organic compounds in study participants versus the National Health and Nutritional Examination Survey III population.

Compound	Mean NHANES III	Mean Study Sample
1,1,1-Trichloroethane	1.780	1.400
1,1-Dichloroethane	0.020	0.020
1,1-Dichloroethylene	0.010	0.010
Tetrachloroethylene	0.710	0.210
Trichloroethylene	0.100	0.070
cis-1,2-Dichloroethylene	0.001	0.012

Table 9 - Correlations between blood, air, and water levels. Continued.

	Number of Observations	Correlation	p[1]
Water with:			
Tenax shower	7	.68	0.09
Household air, omit	6	.09	0.87
Household air, halving	7	.07	0.88
Household air, inclusion	7	.07	0.88
1,1,1-Trichloroethane			
Blood with:			
Tenax shower	7	.00	1.00
Household air, omit	6	.60	.21
Household air, halving	7	.71	.07
Household air, inclusion	7	.36	.43
Water	10	.36	.31
Water with:			
Tenax shower	7	.36	.43
Household air, omit	6	-.26	.62
Household air, halving	7	-.11	.82
Household air, inclusion	7	-.21	.64
1,2 - Dichloroethane			
Water with:			
Tenax shower	1	-	-
Household air, omit	-	-	-
Household air, Halving	-	-	-
Household air, inclusion	-	-	-

[1]p = the probability that the results observed may have occurred by chance.

Table 9 - Correlations between blood, air, and water levels. Continued.

	Number of Observations	Correlation	p[1]
Water with:			
Airbag, shower 10 min	3	.50	0.67
Airbag, shower 20 min	3	1.00	-
Tenax shower	7	.29	0.54
Household air, omit	6	-.37	0.46
Household air, halving	7	-.57	0.18
Household air, inclusion	7	-.57	0.18
Tetrachloroethylene			
Blood with:			
Tenax shower	2	-1.00	-
Household air, omit	6	.66	0.15
Household air, halving	7	.59	0.16
Household air, inclusion	7	.79	0.03
Water	7	-.41	0.35
Water with:			
Tenax shower	0	-	-
Household air, omit	4	-.05	0.94
Household air, halving	5	-.03	0.97
Household air, inclusion	5	.13	0.83
1,1-Dichloroethane			
Blood with:			
Tenax shower	7	-.11	0.81
Household air, omit	6	.90	0.02
Household air, halving	7	.90	0.01
Household air, inclusion	7	.79	0.04
Water	10	.25	0.48

[1]p = the probability that the results observed may have occurred by chance.

Table 9 - Correlations between blood, air, and water levels.

	Number of Observations	Correlation	p[1]
1,1-Dichloroethylene			
Blood with:			
Airbag shower, 10 min	1	-	-
Airbag shower, 20 min	1	-	-
Tenax shower	3	0.50	0.67
Household air, omit	2	-1.00	-
Household air, halving	3	-.50	0.67
Household air, inclusion	3	-.50	0.67
Water	3	1.00	-
Water with:			
Airbag, shower, 10 min	3	0.50	0.67
Airbag, shower, 20 min	3	1.00	-
Tenax shower	5	0.90	0.04
Household air, omit	4	-.63	0.37
Household air, halving	5	-.62	0.27
Household air, inclusion	3	-.62	0.27
cis-1,2-Dichloroethylene			
Blood with:			
Water	2	1.00	-
Trichloroethylene			
Blood with:			
Airbag, shower 10 min	3	-.50	.67
Airbag, shower 20 min	3	-1.00	-
Tenax shower	7	-.54	.22
Household air, omit	6	.26	.62
Household air, halving	7	.54	.22
Household air, inclusion	7	.54	.22
Water	10	-.04	.91

[1]p = the probability that the results observed may have occurred by chance.

Table 8 - Levels of volatile organic compounds measured in the backyard and inside the house for household with backyard sampling.

Compound	Household Sample 1 (6pm - 6am)	Household Sample 2 (6am - 6pm)	Backyard
1,1 - DCE[1]	BLOQ[2]	0.29	ND[3]
T,1-2, DCE[4]	ND	ND	ND
1,1 - DCA[5]	BLOQ	0.12 ppb	BLOQ
1,1,1 - TCA[6]	1.2 ppb	1.5 ppb	1.0 ppb
1,2 - DCA[7]	BLOQ	BLOQ	BLOQ
TCE[8]	0.37 ppb	0.54 ppb	0.12 ppb
PCE[9]	0.22 ppb	0.35 ppb	0.26 ppb

[1]1,1 - Dichloroethylene

[2]Below the limit of quantitation

[3]Not detected

[4]Trans - 1,2 - Dichloroethylene

[5]1,1 - Dichloroethane

[6]1,1,1 - Trichloroethane

[7]1,2 - Dichloroethane

[8]Trichloroethylene

[9]Tetrachloroethylene

Table 7 - Descriptive statistics for air measurements.

Variable	Number of Measurements	Mean[1]	Median[1]	Range[1]
Airbag, 0 min				
1,1-Dichloroethylene	0	-	-	-
Trichloroethylene	0	-	-	-
Tetrachloroethylene	0	-	-	-
Airbag, 10 min				
1,1-Dichloroethylene	3	43.60	54.80	17.60-58.40
Trichloroethylene	3	68.72	68.17	55.20-82.80
Tetrachloroethylene	0	-	-	-
Airbag, 20 min				
1,1-Dichloroethylene	3	50.00	49.70	20.20-80.10
Trichloroethylene	3	66.33	72.60	34.60-91.80
Tetrachloroethylene	0	-	-	-
Shower, Tenax tubes				
1,1-Dichloroethylene	7	7.83	6.66	1.29-25.11
1,1-Dichloroethane	7	13.70	9.14	3.79-30.62
1,1,1-Trichloroethane	7	24.65	23.37	8.61-47.12
1,2-Dichloroethane	1	2.31	-	-
Trichloroethylene	7	10.95	10.44	5.36-18.71
Tetrachloroethylene	2	1.83	1.83	1.67-1.95
Household-averaged by omission	6	0.18	0.14	0.06-0.50
1,1-Dichlorethylene				
1,1-Dichloroethane	6	0.18	0.10	0.06-0.40
1,1,1-Trichloroethane	6	1.31	1.35	0.50-1.95
1,2-Dichlorethane	0	-	-	-
Trichloroethylene	6	0.31	0.34	0.01-0.46
Tetrachloroethylene	6	0.19	0.16	0.06-0.40
Household-averages by halving				
1,1-Dichloroethylene	7	0.17	0.10	0.06-0.50
1,1-Dichloroethane	7	0.17	0.10	0.06-0.40
1,1,1-Trichloroethane	7	1.24	1.33	0.50-1.95
1,2-Dichloroethane	0	-	-	-
Trichloroethylene	7	0.38	0.41	0.10-0.81
Tetrachloroethylene	7	0.19	0.18	0.06-0.40

[1]In parts per billion

Table 6 - Descriptive statistics for water measurements.

Variable	Number of Measurements Used for Analysis	Mean[1]	Median[1]	Range[1]
1,1-Dichloroethylene	7	20.64	2.80	0.80-62.6
1,1-Dichloroethane	10	34.69	33.35	8.00-75.3
Chloroform	3	10.73	11.20	7.00-14.0
1,2-Dichloroethane	5	5.79	2.53	1.86-13.60
1,1,1-Trichloroethane	10	211.93	191.30	83.50-400.00
Trichloroethylene	10	42.05	40.85	20.70-65.60
1,1,2,2-Tetrachloroethane	3	21.17	4.20	3.20-56.10
Tetrachloroethylene	7	1.86	0.80	0.50-6.60
cis-1,2-Dichloroethylene	3	76.73	93.00	42.60-94.60
trans-1,2-Dichloroethylene	1	1.14	1.14	-

[1]In parts per billion

Note: When half or more measurements of a given variable were an indication either that no chemical had been detected or that the chemical was present but at a level that could not be quantified, only numerical values were included in analyses. When fewer than half of the measurements were below the detection limit, the measurements reported as indications that no chemical had been detected were assigned a zero value, and the measurements reported as indications that the chemical was present but at a level that could not be quantified were assigned a value equal to half the limit.

Table 5 - Descriptive statistics for blood measurements.

Variable	Number of Measurements Used for Analysis	Mean[1]	Median[1]	Range[1]
1,1,1-Trichloroethane	10	1.40	1.40	0.45-1.99
1,1-Dichloroethane	10	0.02	0.02	0.01-0.03
1,1-Dichloroethylene	4	0.01	0.01	0.01-0.02
cis-1,2-Dichloroethylene	5	0.01	0.01	0.00-0.03
Tetrachloroethylene	10	0.21	0.17	0.12-0.65
Trichloroethylene	10	0.07	0.05	0.01-0.30

[1]In parts per billion

Note: When half or more measurements of a given variable were an indication either that no chemical had been detected or that the chemical was present but at a level that could not be quantified, only numerical values were included in the analyses. When fewer than half of the measurements were below the detection limit, the measurements reported as indications that no chemical had been detected were assigned a zero value, and the measurements reported as indications that the chemical was present but at a level that could not be quantified were assigned a value equal to half the limit.

Table 4 - Numbers of observations, nondetects, and below the detection limits. Continued.

Variable	Number of Observations	Number of Nondetects	Number Below Detection
Household, Second 12 Hours			
1,1 - Dichloroethylene	6(1)	0(0)	2(0)
trans-1,2 - Dichloroethylene	6(1)	6(1)	0(0)
1,1 - Dichloroethane	6(1)	0(0)	2(0)
1,1,1 - Trichloroethane	6(1)	0(0)	0(0)
1,2 - Dichloroethane	6(1)	1(0)	5(1)
Trichloroethylene	6(1)	0(0)	0(0)
Tetrachloroethylene	6(1)	0(0)	3(0)

Table 4 - Numbers of observations, nondetects, and below the detection limits. Continued.

Variable	Number of Observations	Number of Nondetects	Number Below Detection
Airbag, 10 Min			
1,1 - Dichloroethylene	3(1)	0(0)	0(1)
trans - 1,2 - Dichloroethylene	3(1)	0(1)	3(0)
Trichloroethylene	3(1)	0(0)	0(0)
Tetrachloroethylene	3(1)	3(0)	0(1)
Airbag, 20 Min			
1,1 - Dichloroethylene	3	0	0
trans - 1,2 Dichloroethylene	3	0	3
Trichloroethylene	3	0	0
Tetrachloroethylene	3	3	0
Shower, Tenax			
1,1 - Dichloroethylene	7(5)	0(0)	1(1)
trans - 1,2 - Dichloroethylene	7(5)	6(4)	1(1)
1,1 - Dichloroethane	7(5)	0(0)	0(0)
1,1,1 - Trichloroethane	7(5)	0(0)	0(0)
1,2 - Dichloroethane	7(5)	0(0)	7(4)
Trichloroethylene	7(5)	0(0)	0(0)
Tetrachloroethylene	7(5)	0(0)	6(4)
Household, First 12 Hours			
1,1 - Dichloroethylene	7	0	4
trans - 1,2 - Dichloroethylene	7	7	0
1,1 - Dichloroethane	7	0	4
1,1,1 - Trichloroethane	7	0	0
1,2 - Dichloroethane	7	2	5
Trichloroethylene	7	0	1
Tetrachloroethylene	7	0	2

Table 4 - Numbers of observations, nondetects, and below the detection limits.

Variable	Number of Observations	Number of Nondetects	Number Below Detection
Blood			
1,1,1 - Trichloroethane	10	0	0
1,1 - Dichloroethane	10	0	0
1,1 - Dichloroethylene	10	6	0
cis - 1,2 - Dichloroethylene	9	4	0
trans - 1,2 - Dichloroethylene	9	9	0
Tetrachloroethylene	10	0	0
Trichloroethylene	10	1	0
Water			
1,1 - Dichloroethylene	7	0	0
1,1 - Dichloroethane	10	0	0
Chloroform	7	4	0
1,2 - Dichloroethane	10	5	0
1,1,1 - Trichloroethane	10	0	0
Trichloroethylene	10	0	0
1,1,2,2 - Tetrachloroethane	7	4	0
Tetrachloroethylene	7	3	0
cis - 1,2 - Dichloroethylene	3	0	0
trans - 1,2 - Dichloroethylene	10	7	2
Air			
Airbag, 0 Min			
1,1 - Dichloroethylene	3(1)[1]	3(1)	0(0)
trans - 1,2 - Dichloroethylene	3(1)	3(1)	0(0)
Trichloroethylene	3(1)	2(0)	0(0)
Tetrachloroethylene	3(1)	3(1)	0(0)

[1]Parentheses indicate duplicate samples.

Table 3 - Levels of trichloroethylene and total volatile organic compounds, study participants versus study nonparticipants.

Trichloroethylene

	Range	Mean	p[1]
Participants	21 - 76 ppb	45 ppb	0.74
Nonparticipants	11 - 120 ppb	48 ppb	

Total Volatile Organic Compounds

	Range	Mean	p[1]
Participants	190 - 740 ppb	350 ppb	0.56
Nonparticipants	150 - 610 ppb	320 ppb	

[1]p = the probability that the results observed may have occurred by chance.

Table 2 - Reasons for excluding households.

Reason	Number of households
No contact	3
Refusal	3
Well not used for drinking or cooking	18
Water filter in use	5
No nonpregnant women aged 20 to 65	10
Women of household work outside the home more than 25 hours per week	14
Total	53

Table 1 - Water levels of volatile organic compounds in southeast Rockford as of October, 1989.

Compound[1]	Frequency[2]	Mean[3]	Range[4]	EPA DWEL[6]
PCE	53	1.2	Tr[5] - 140	680
TCE	87	22	Tr - 140	260
TCA	92	69	Tr - 430	1000
t-1,2-DCE	49	2.7	Tr - 100	350
C-1,2-DCE	4.0	1.5	6.0 - 110	350
1,2-DCA	13	0.8	0.7 - 14	-
1,1-DCE	78	66	0.1 - 240	350
1,1,-DCA	74	17	Tr - 100	-
CH_2Cl_2	1.0	6.0	3.0 - 9.0	-
$CHCl_3$	22	1.4	0.7 - 14	-
$CHBr_3$	0.5	3.6	-	-
Benzene	0.5	6.5	-	-

[1]PCE - Tetrachloroethylene
TCE - Trichloroethylene
TCA - 1,1,1-Trichloroethane
t-1,2-DCE - Trans-1,2,-Dichloroethylene
c-1,2-DCE--cis-1,2-Dichloroethylene
1,2,-DCA - 1,2- Dichloroethane
1,1-DCE - 1,1,-Dichloroethylene
1,1-DCA - 1,1,Dichloroethane
CH_2Cl_2 - Methylene Chloride
$CHCl_3$ - Chloroform
$CHBr_3$ - Bromoform

[2]Frequency of Detection - percentage of time a compound was found in private well samples.

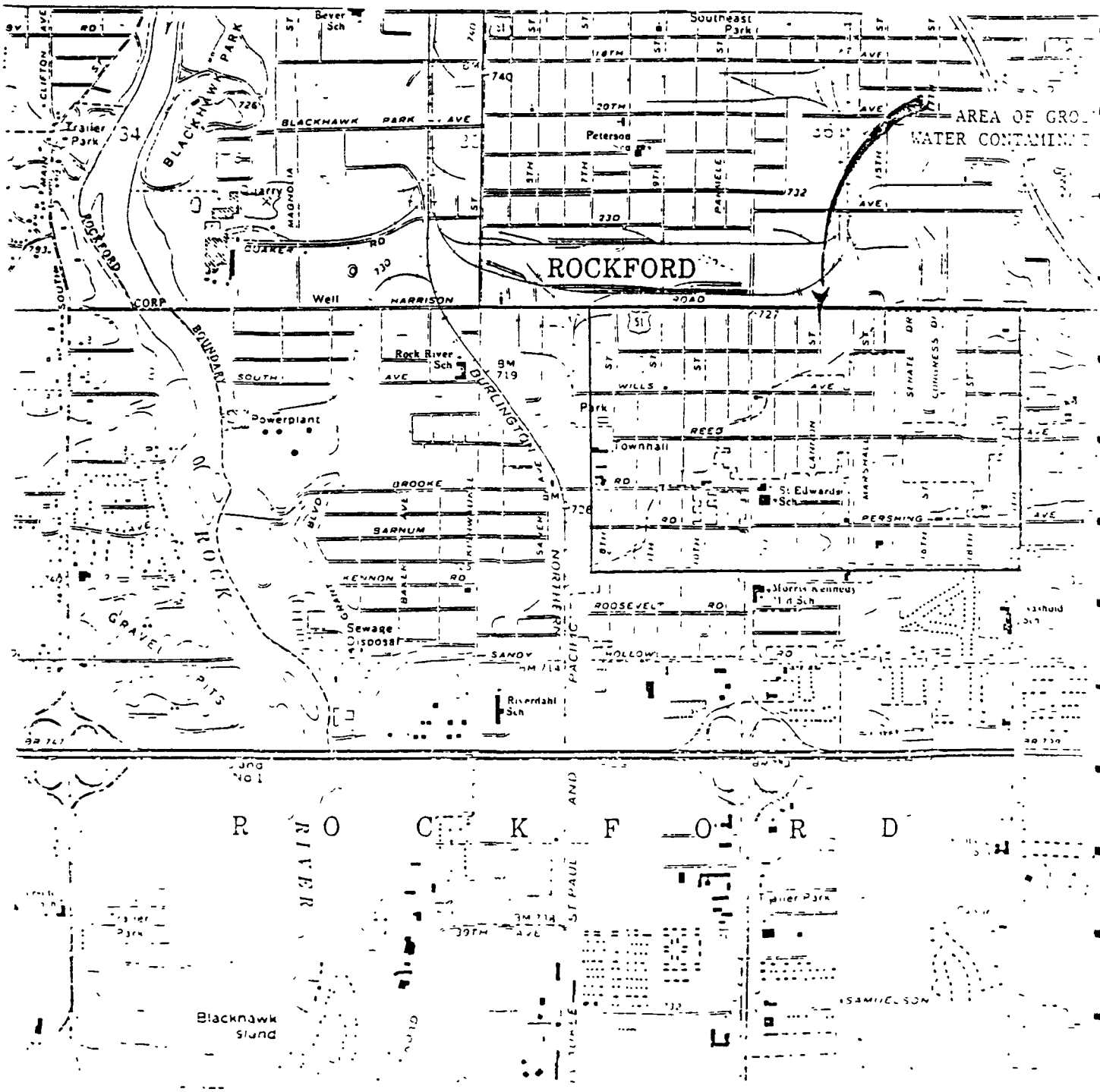
[3]Mean Concentration - arithmetic mean of all sample results including non-detects (counted as zero) in parts per billion (ppb).

[4]Range of Concentration - range of positive detections excluding non-detects in ppb.

[5]Tr - trace. Limit of detection is reported as 0.1 ppb.

[6]Environmental Protection Agency Drinking Water Equivalent Level.

FIGURE 1 - USGS 7.5 Minute Series Map of
Southeast Rockford. 1976



1.0 SCOPE OF APPLICATION

Tenax tube sampling is utilized to identify specific contaminants in air. Compounds that can be determined by Tenax/CMS air tubes are nonpolar organics having boiling points in the range of approximately 80^{0}-100^{o}C. However, not all compounds falling into this category can be determined. Table 1 (13.0 Appendices) lists compounds for which ERT/TAT analyzes Tenax/CMS. Analysis is performed by thermal desorption into a gas chromatograph/mass spectrometer/data system (GC/MS/DS).

2.0 METHOD SUMMARY

Tenax tube sampling is performed by drawing a known volume of air through a Tenax-carbonized molecular sieve (CMS) adsorbent. Volatile organic compounds are captured on the adsorbent while major inorganic atmospheric constituents pass through (or are only partially retained). After sampling, the tube is returned to the laboratory for analysis (EPA Method TO1).

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Tenax tubes contain a granular inert chemical compound with adsorbent properties. A flame-sealed outer glass tube protects the tube from contamination. This outer glass tube must be broken prior to sampling. The Tenax/Carbonized Molecular Sieve (CMS) air tube is 6.0 mm O.D. and 4 mm I.D. containing two sections of 150 mg Tenax 35/60 mesh and 150 mg CMS 60/80 mesh.

Prior to site work, the culture tube is cleaned and prepared for field use.

1. A plug of precleaned silanized glass wool (methanol rinsed, baked in an oven @ 120^{o}C) is placed in the bottom of each tube.

2. The culture tubes are placed in an oven for at least 2 hours at 120^{o}C. The Teflon lined caps are not baked.

3. The culture tubes are removed from the oven and allowed to cool. The tubes are then purged with high purity air or preferably, high purity nitrogen.

4. The culture tubes are placed in a zip lock bag or whirl pack, then in a clean metal paint can containing activated charcoal and desiccant. The paint can is sealed until use in field.

5. Paint cans should contain approx. 1/2 inch of activated charcoal and 2 to 3 vials of activated desiccant. Charcoal and desiccant are activated by placing in an oven at 120°C for a least 2 hours or until the desiccants turns from pink to dark blue color. The paint cans should be previously purged with high purity air or nitrogen, then sealed.

6. Refrigerate the samples and keep out of sunlight. Storage for more than 4 weeks is not recommended.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Contamination of the Tenax/CMS air tubes with the compound(s) of interest is a common problem. To minimize this problem, the user must be extremely careful in the preparation, storage, and handling of the air tube throughout the sampling and analysis process. To avoid contamination from skin oils, a lint-free glove must be used when handling Tenax air tubes.

5.0 EQUIPMENT

5.1 Equipment List

Gilian Personal Sampling Pump
Dual Rotometer with stand and precalibrated flow rate
Tenax Tube, preferably of the same lot number
Flexible Tygon Tubing (for attaching the tube holder system to the suction side of the pump)
Universal Tube Holder System
- o Dual variable manifold flow controller
- o Tube holder end with rubber boot adaptor
- o Sleeves - clear plastic housings

Glass Cracker
Lint Free Cloth
Glass Wool
Teflon Tape
1-gallon paint can
Culture Tubes
Carbon
Desiccant

5.2 Equipment Sources

Presently REAC obtains Tenax from Supelco Inc., Bellefonte, PA, at (800) 247-6628; Technical Service (814) 359-3441.

6.0 REAGENTS

Methanol is used in the lab to clean the glass tubing which holds the Tenax samples. This is performed prior to site work.

7.0 PROCEDURES

7.1 Calibration Procedures

Assemble the calibration train as shown in Figure 1 using a rotometer, sampling pump, manifold, and representative Tenax tubes. The same lot # Tenax tube is used for both sampling and calibrating.

1. Adjust the sampling pump to the low flow mode.
2. Remove the cap ends on the flow controller manifold. To adjust flow, turn the needle valve with a small screw driver (counter-clockwise to increase, clockwise to decrease).
3. Turn the flow adjust screw on each manifold until the float ball on the rotometer is lined up with the precalibrated flow rate value. A sticker on the rotometer should indicate this value.
4. Affix a sticker to the manifold and pump indicating the calibrated flow rate and media.
5. Remove the representative Tenax tubes from the sleeves.

The pump and manifold (including boots) are calibrated as a unit and should not be separated until the samples have been collected.

The pump and manifold are calibrated on-site in the clean zone immediately prior to the sampling collection. See Attachment 1 for flow rate ranges.

7.2 Field Operation

1. Calibrate pumps.
2. Crack the outer glass tube using a glass cracker.
3. Use a clean, lint-free cloth or gloves, to remove the Tenax tube from the outer glass housing.
4. Insert the Tenax tube into a boot, with the dark charcoal section closest to the manifold.
5. Attach a sleeve onto the boot. Do not enclose the Tenax tube end.
6. Connect the tubes to a double manifold.
7. Set up the sampling train, by attaching one end of the Tygon tubing (approx. 2 feet) to the manifold; and the other end to the inlet plug on the pump See Figure 2.

8. Place the sampling tube in the breathing zone. The pump and tube can be placed on a drum or hooked to a fence. A wooden dowel can be used to hold the pump.
9. Place the tube either vertical or horizontal.
10. Adjust the time on the pump first. Turning past the zero mark several times.
11. Turn the pump on.
12. Record weather data (e.g., ambient temperature, barometric pressure, relative humidity and wind direction).
13. Check the pump at the midpoint of the sampling period if longer than 4 hours.

7.3 Post Procedures

1. At the end of the sampling period, check the fault button to obtain pump sampling time.
2. Record the run time (this indicates whether or not the pump ran full).
3. Check the flow rates and record the values.
4. Remove the Tenax tubes from sleeves using a lint free cloth.
5. Place the Tenax tube in a culture tube. Tenax tubes from the same manifold can be placed in the same culture tube provided the flow rates are the same.
6. Place a sample sticker indicating sample ID# on the culture tube. Do not put a sample sticker on the Tenax tube itself as this will contaminate the tube.
7. Wrap the inside threads of the culture tube with Teflon tape. Screw culture tube lid on and tape over.
8. Place the culture tubes into a zip lock bag or a whirl pack, then into a clean 1-gallon metal paint can containing desiccant and activated charcoal.
9. Keep the samples refrigerated and out of sunlight. Storage for more than 4 weeks is not recommended.
10. Indicate all applicable information on the chain of custody, (e.g., sample volume, sample ID#).
11. Provide a copy of the air data sheets and the analytical methodology with the samples to the lab.

8.0 CALCULATIONS

The flow rate times the total sample time yields the total volume for that sample.

Flow Rate x Time (minutes) = total volume

The volumes for each sample should be indicated on the chain of custody.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

Varying the sample volumes at the same location provides field QA/QC.

1. Provide 1 appropriately labeled field blank per sampling period. Handle this tube in the same manner as the sampling tube (break, seal, and transport) except that no air is sampled through this tube.
2. Provide several appropriately lot blank tubes. These tubes are taken directly from the Tenax tube box. Do not break the outer glass housing. Place in a zip lock bag and keep with other samples. Indicate the lot blank number on the Chain of Custody Form.

10.0 DATA VALIDATION

Results of the quality control samples (log and trip blanks) will be evaluated for contamination. This information will be utilized to qualify the environmental sample results accordingly with data quality objectives.

Data will be qualified accordingly with acceptable variation on the prescribed flow rates (see Table 2).

11.0 HEALTH AND SAFETY

Prior to initiating survey activities, an analysis of risk is required to determine the hazards posed to sampling personnel. This will estimate any potential exposures to personnel, and define the extent of safety planning needed to complete the task. Depending upon the hazards identified, a safety plan may be required to prior to performing any site entry. In addition, real time monitoring may be necessary in order to verify ambient conditions and adequate respiratory protection.

Specific hazards associated with Tenax tube sampling include:

1. Sharp edges associated with broken glass tubes where the Tenax tubes are stored.

2. Small pieces of glass flying during "cracking" of the tube.

3. Sharp blades used when cutting the tubing.

4. Access to sampling locations.

12.0 REFERENCES

U.S. EPA, Compendium of Methods for Determination of Toxic Organic Compounds in Ambient Air, EPA 600/4-84/041, December, 1984.

U.S. EPA, Characterization of Hazardous Waste Sites - A Methods Summary: Volume II, Available Sampling Methods, 2nd Edison, EPA 600/4-84/76, December, 1984.

TABLE 1. AIR ANALYSIS BY GC/MS

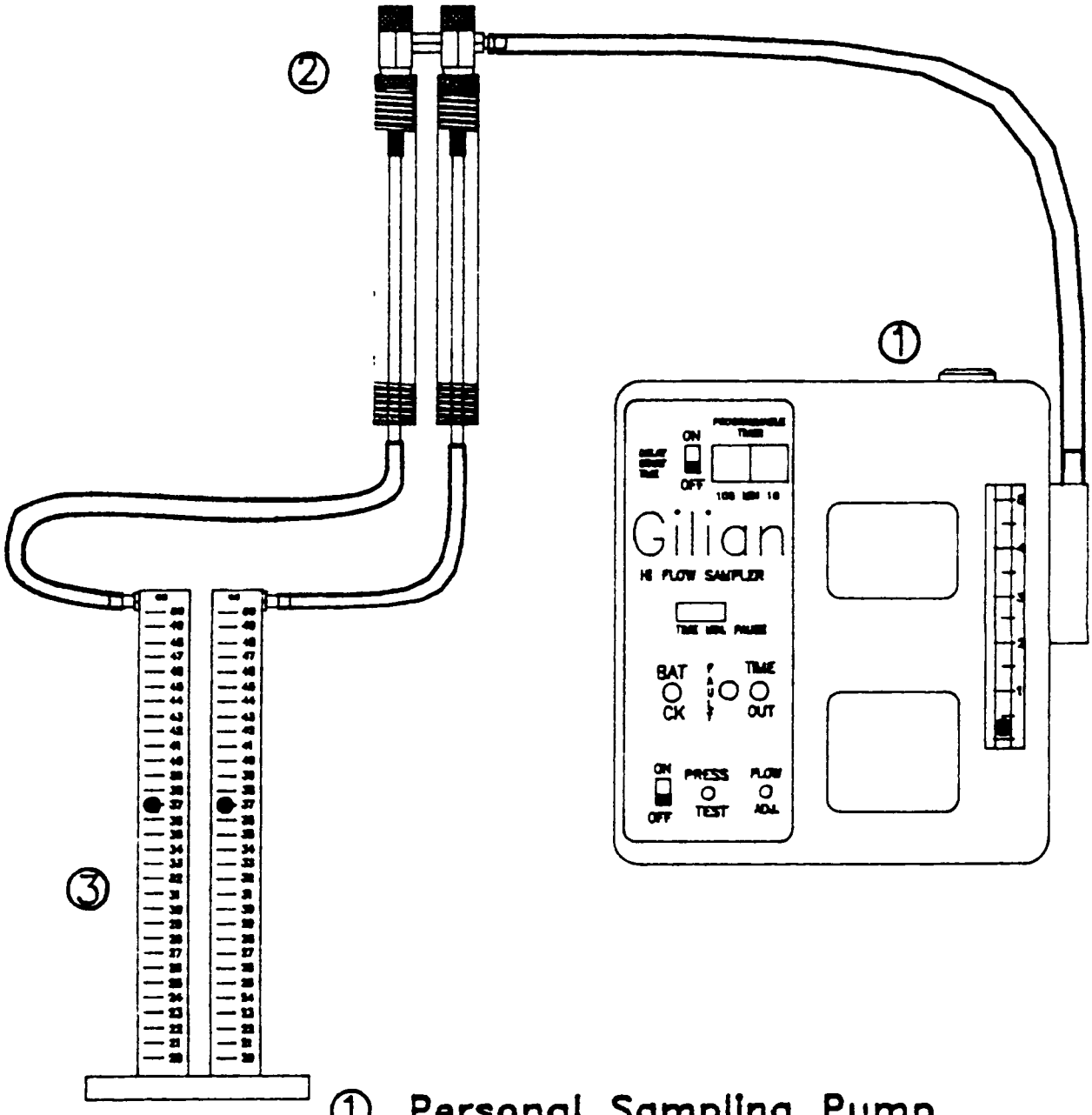
Compound	Safe Sampling Volume (l)
1) # Vinyl Chloride	--
2) # Trichlorofluoromethane	--
3) # 1,1-Dichloroethene	1.05
4) # Methylene Chloride	--
5) # Trans-1,2-Dichloroethene	4.05
6) # 1,1-Dichloroethane	1.05
7) # Bromochloromethane*	--
8) # 1,1,1-Trichloroethane	2.85
9) # Carbon Tetrachloride	4.65
10) # Benzene	4.65
11) # 1,2-Dichloroethane	4.05
12) # Trichloroethylene	4.2
13) # Toluene	28.50
14) # Tetrachloroethylene	36.0
15) # Ethylbenzene	--
16) # Meta-Xylene	225.0
17) # Ortho-Xylene	225.0
18) # Styrene	225.0
19) # p-Bromofluorobenzene*	--
20) # Meta-Ethyltoluene	--

* Surrogate - Surrogates are injected into the Tenax to determine adsorption efficiencies.

TABLE 2. RECOMMENDED FLOW RATES AND SAMPLE VOLUMES

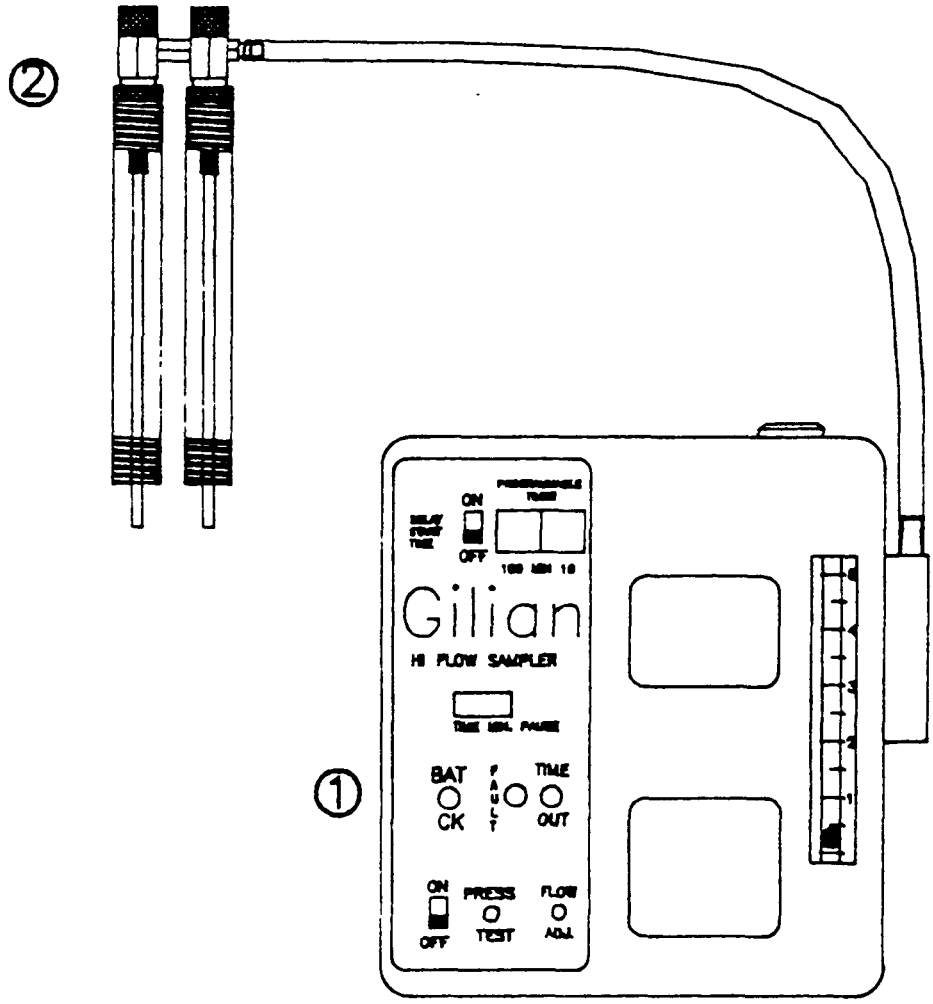
Flow Rates	
Maximum	100 cc/min
Optimal	50 cc/min
Minimum	20 cc/min
Volumes	
Maximum	5 l
Optimal	2 l
Minimum	.5 l

Diagram 1 - Tenax Calibration with a Rotometer



① Personal Sampling Pump
② Tenex Tube with Double Manifold
③ Double Rotometer

Diagram 2 - Tenax Sampling Train



① Personal Sampling Pump
② Tenex Tube with Double Manifold

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1.0 SCOPE AND APPLICATION

Charcoal tube sampling is utilized to identify specific contaminants in air. The greatest selectivity of activated charcoal is towards nonpolar organic solvent vapors, (e.g., carbon tetrachloride, chlorobenzene and toluene). Organic compounds that are gaseous at room temperature, reactive, polar, or oxygenated (aldehyde alcohols and some ketones) are either not adsorbed (relatively early breakthrough) or inefficiently desorbed. Prior to sampling, the entire sampling train (rotometer, sampling pump, manifold, and charcoal tube) is calibrated for flow rate.

2.0 METHOD SUMMARY

Charcoal tube sampling is performed by drawing a known volume of air through a charcoal adsorption tube. As air is drawn through the tube during sampling, gases and vapors are adsorbed onto the surface of the charcoal. After sampling the tubes are delivered to the laboratory for analysis.

3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

Charcoal used for sampling is housed in a glass tube that has been flame sealed. Charcoal tubes most often used contain 150 mg and 600 mg of charcoal. The smaller 150-mg tube is 7-cm long with a 6-mm ID and a 4-mm OD containing 2 sections of 20/40 mesh activated charcoal separated by urethane foam. The adsorbing section contains 100-mg of charcoal, the backup section 50-mg of charcoal. The larger 600-mg tube is 11-cm long with a 8-mm ID and a 6-mm OD containing 2 sections of 20/40 mesh activated charcoal separated by urethane foam. The adsorbing section contains 400 mg of charcoal, the backup section contains 200-mg of charcoal.

To preserve and store sample:

1. Place the sample in the whirl bag. If duplicate samples have been collected, place both tubes in one whirl bag.
2. Indicate all applicable information on the Chain of Custody Form, (e.g., sample volume, ID #, location, date, and weather parameters).
3. If the sample tube must be stored for more than a week, refrigeration is recommended.
4. Provide analytical methodology with the sample to the lab.

4.0 INTERFERENCES AND POTENTIAL PROBLEMS

High temperature and humidity, and low sampling flow rates may cause a decrease in the adsorption capacity of activated carbon.

5.0 EQUIPMENT

5.1 Equipment List

Gilian Personal Sampling Pump
Dowel Rods
Single or Dual Rotometer with stand and desired precalibrated flow rate
Carbon Tubes - (600 mg or 150 mg)
Flexible PVC Tubing - (for attaching the tube holder system to the suction side of the pump)

Universal Tube Holder System
- o Sleeves (or support tubes to hold tubes in place)
- o Single or dual manifold flow controller
- o Tube holder end and hose barbs (Tube holder ends support and seal the sampling tube within the plastic housing. Hose barbs connect tube holders to the tubing attached to the suction side of the pump)
- o Glass Cracker
- o Zip Lock Bag
- o Whirl Bags
- o Plastic Caps

5.2 Equipment Source

Tubes are commercially available from SKC, Inc. and from Mine Safety Appliance Co., both of Pittsburgh, Pennsylvania.

SKC: 1-800-752-8472
Mine Safety Appliance Co.: 1-800-MSA-2222

6.0 REAGENTS

This procedure utilizes totally dedicated equipment and does not require reagents.

7.0 PROCEDURES

7.1 Calibration Procedures

To save time, sampling pumps can be calibrated in the office prior to arriving at the site. For projects which require strict QA/QC, the calibration must be checked in the field prior to, and upon completion of, sampling.

Assemble the calibration train as shown in Diagram 1, using a rotometer, sampling pump, manifold (low flow only) and representative charcoal tube. Use the same lot # charcoal tube for both sampling and calibrating.

1. Depending on the flow rate, adjust the sampling pump to the low or high flow mode (high flow > 750 cc/min).
2. For low flow calibration, turn the flow adjust screw on the manifold until the float ball on the rotometer is aligned with the precalibrated flow rate value. A sticker on the rotometer should indicate this value.
3. Affix a sticker to the manifold and pump indicating flow rate and media.
4. Remove the representative charcoal tube from the sleeve. The pump and manifold are calibrated as a unit and should not be separated until the samples have been collected. If the charcoal tube is run straight without a manifold, the calibration is performed by adjusting the flow directly on the pump.

7.2 Field Operation

1. Mobilize to the clean zone and perform calibration.
2. Mobilize to the sampling location.
3. Crack the charcoal tube ends using a glass cracker.
4. Insert the charcoal tube in the sleeve with arrow pointing in the direction of air flow (the smaller section is used for a backup and is positioned nearest the sampling pump).
5. Screw the tip onto the sleeve so the charcoal tube is held in place.
6. Attach the sleeve(s) to a single or double manifold. At higher flow rates (>750 cc/min), charcoal tubes can run straight without a manifold.
7. To set up the sampling train, attach one end of the tygon tubing (approx. 2 foot) to the tip of the sleeve or manifold. Attach the other end of the tubing to the inlet plug on the pump. See Diagram 2.
8. Adjust time on pump by adjusting past the zero mark several times to erase the preprogrammed time.
9. Place the charcoal tube in a vertically on a dowel rod.
10. Record weather data (i.e., ambient temperature, barometric pressure, relative humidity, and wind direction).
11. Turn the pump on.
12. After the pump has run full, check the fault button to obtain the sampling time (this will indicate whether or not the pump ran full).

7.3 Post Procedures

1. Record the sampling time.
2. Remove the charcoal tube from the sleeve.
3. Capped charcoal tubes with plastic caps immediately after sampling. Never use rubber caps.
4. Place a sample ID # label on the tube.
5. Place the sample in a whirl bag labeled with sample ID#, total volume, and required analysis. If duplicate samples have been collected, place both tubes in one whirl bag.
6. Indicate all applicable information on the Chain of Custody Form (e.g., sample volume, ID #, location, date, and weather parameters).
7. If the sample tube must be stored for more than a week, refrigeration is recommended.
8. Provide analytical methodology to the lab with the samples.

REAC uses NIOSH Methods 1501, Aromatic Hydrocarbons; 1500, Hydrocarbons BP 36°-126°C; and 1003, Halogenated Hydrocarbons for the analysis of the charcoal tubes. Other analytical parameters may be required. The Task Leader should check with the ERT Work Assignment Manager for the appropriate analytical methodology.

8.0 CALCULATIONS

The total volume of a sample is calculated by multiplying the total sample time by the flow rate. The total volume for each sample should be indicated on the chain of custody.

9.0 QUALITY ASSURANCE/QUALITY CONTROL

1. Provide 1 field blank per sampling period or 22 field blanks for every 10 samples whichever is greater. The tube should be handled in the same manner as the sampling tube (break, seal, and transport) except that no air is sampled through this tube.

2. Provide several appropriately labeled lot blank tubes. The lab analyzing the samples can better determine the number of lot blank tubes required. These tubes are taken directly from the charcoal tube box. Do not break the ends.

10.0 DATA VALIDATION

Results of the quality control samples (lot and trip blanks) will be evaluated for contamination. This information will be utilized to qualify the environmental sample results accordingly with data quality objectives.

11.0 HEALTH AND SAFETY

Prior to initiating survey activities, a risk analysis is required to determine the hazards posed to sampling personnel. This will estimate any potential exposures to personnel, and define the extent of safety planning needed to complete the task.

Depending upon the hazards identified, a safety plan may be required to prior to performing any site entry. In addition, real time monitoring may be necessary in order to verify ambient conditions and adequate respiratory protection.

Specific hazards unique to charcoal tube sampling include:

1. Sharp edges associated with the tubes after they have been "cracked."

2. Sharp blades used to cut sampling tubing.

3. Walking and working surfaces, as well as possible heights, associated with access to sampling locations.

12.0 REFERENCES

Eiler, Peter M., ed. NIOSH Method 1003, Halogenated Hydrocarbons. In: NIOSH Manual of Analytical Methods, Third Edition, U.S. Gov.'t Printing Office, Washington, D.C., 1987. p. 1003-1.

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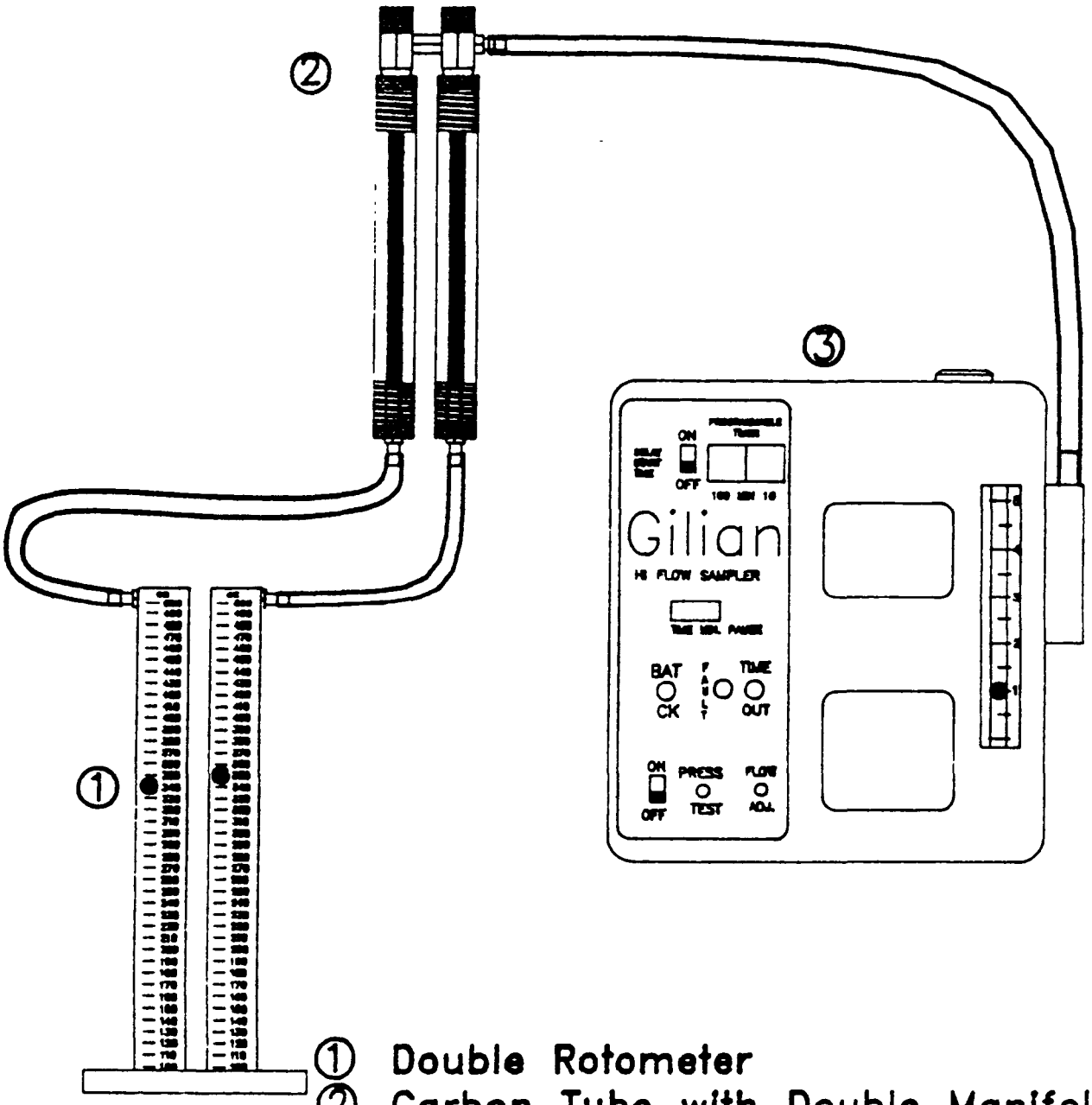
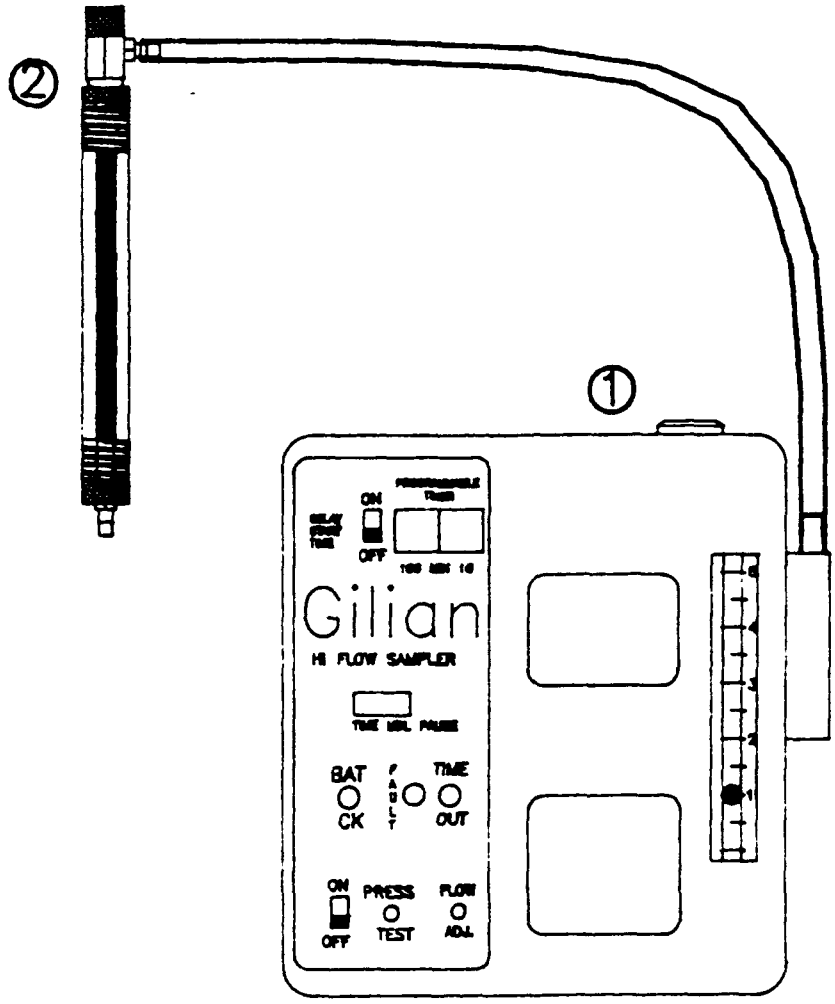
Diagram 1 - Calibrating a Double Manifold Charcoal Tube with a Rotometer



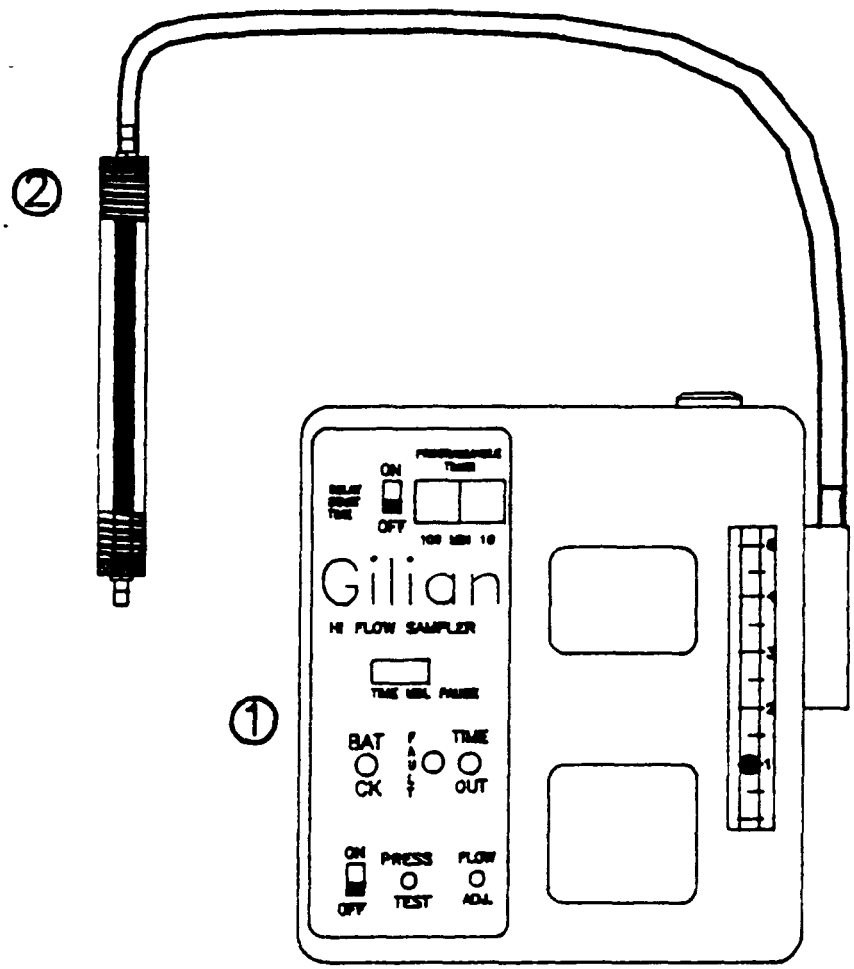
① Double Rotometer
② Carbon Tube with Double Manifold
③ Personal Sampling Pump

Diagram 2 - Charcoal Sampling, Single Manifold



① Personal Sampling Pump
② Carbon Tube Single Manifold (600mg or 150mg)

Diagram 3 - Carbon Sampling, Straight



① Personal Sampling Pump
② Carbon Tube – Straight